Coproducts and Near Coproducts of Fuel Ethanol Fermentation from Grain

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by

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Executive Summary

The technology utilized for the conversion of plant materials to alcohol is both ancient and well developed. However, there is room for improvement, both in process technology and coproduct utilization. Greater exploitation of byproducts would lead to enhanced efficiency and greater

economic profitability for ethanol producers and for farmers. The establishment of markets for ethanol coproducts and near coproducts will ensure the future success of the ethanol industry.

Because, in many cases, the utilization of coproducts comes about mainly as a waste disposal solution, coproducts are not always utilized to their maximum potential. In most instances, fuel ethanol byproducts are used in the animal feed industry. If products for other markets can be developed, there is a greater potential for profitability. New biotechnological techniques are being developed that will enable the production of new materials for the food, cosmetic and pharmaceutical industries.

Biotechnology research in Canada is highly developed. However, some research projects with commercial potential have not been brought to the marketplace because work has terminated at the bench. In other instances research has been duplicated. A reference database has been developed to aid researchers in becoming familiar with the work that has been done concerning the use of byproducts formed when grain is fermented to produce fuel ethanol. This database is available on the Internet at http://res2.agr.ca/research-recherche/cfar/coproduc.html.

In this report, the four major small grain cereals (corn, wheat, oats and barley) were investigated. A summary of the current scientific and technical literature related to coproducts and near-coproducts of fuel ethanol produced from these grains was completed. Use as animal feed was not covered, the focus being on higher value utilization.

The type of coproducts produced depends on the particular feedstock utilized and on the processing technology used. Starch is consumed in the fermentation to produce ethanol, while protein and fibre form the major byproducts. Protein has been investigated as both a concentrate for human food and as an industrial material. Cereal fibre has generated a great deal of interest in recent years as it has become linked to dietary health effects.

Other minor components have also been considered for extraction before or after the fermentation process. Additionally, specific byproducts such as ethanol stillage have been appraised for use as media for microbial production of value-added compounds including organic acids and pigments.

A study was done of current markets for cereal derivatives in order to assess the demand for potential coproducts and near coproducts of ethanol production. Awareness of how cereal components are now being used allows industry and researchers to direct their efforts towards potential commercial interests.

Cereal derivatives are used in the food, cosmetics and pharmaceutical industries. Particular components have been found to have significant health effects when included in the diet of persons suffering from ailments such as heart disease, diabetes and cancer. The development of functional food or nutraceutical markets may play a significant role in the future.

Different components of the small grain cereals are used in a number of cosmetics, toiletries and pharmaceutical products. These range from proteins and their building blocks, amino acids to fats, fibres, vitamins and other minor components. If cost-effective extraction methods can be developed, certain derivatives may have the potential to replace synthetically produced compounds currently in use. This will particularly serve the "green" or bio-based chemical market which has grown significantly in recent years. Development of niche or novel markets, rather than those that are already highly competitive and adequately served, offer the greatest window of opportunity.

New ethanol plants under development in Canada are well aware of the need to fully exploit all components of the cereal grain used as a feedstock. While ethanol, for fuel and/or industrial usage, will continue to be a major output, fuel ethanol producers are looking at generating a number of product streams to diversify their profit potential. Preprocessing technologies to separate specific grain fractions before fermentation are being considered.

Current research efforts by industry, universities and governments have been summarized. This information was gathered from a variety of sources including electronic research databases (Inventory of Canadian Agri-Food Research (ICAR), Current Research Information System (CRIS, U.S.), Australian Rural Research in Progress (ARRIP), Agricultural Research Projects (AGREP, European Union) and Crop Association Sponsored Research Archive (CASRA, U.S.), conference proceedings, current publications and contact with industry, university and government research facilities. Articles were prepared and appeared in two publications, Biomass & Bioenergy Innovations (formerly Bioenergy West) and The Energy Independent, publicizing this project and asking for contributions of information.

A major effort in the area of coproduct research is in progress in the United States where the fuel ethanol industry is much more extensive than in Canada. In the U.S., research into corn coproducts predominates, corn being the most common ethanol feedstock by far. Many aspects of corn-to-ethanol production are being investigated in relation to coproduct production including genetic modification of corn cultivars, process technology, extraction procedures, new industrial applications and further value-added processing of byproducts such as corn fibre.

In Canada, research directly into coproducts of ethanol fermentation from small grain cereals is minimal. However, research into value-added processing and utilization of minor cereal components, particularly from wheat, oats and barley, is receiving a great deal of attention. Some of the discoveries that have been made in the laboratory can be applied in an ethanol plant to produce value-added coproduct streams and indeed there are plans to do this in the near future. At present, investigations into insoluble and soluble fibre fractions appear to have the most potential for commercialization.

The fuel ethanol industry appears to be on the threshold of new direction in Canada. Development of the industry has the capacity to improve the sustainability of rural communities, provide economic opportunities in the agri-food sector and to preserve the environment. However, fuel ethanol production is not profitable on a stand-alone basis. There is a need to move towards cereal grain biorefineries that produce ethanol as only one of a number of coproduct streams including foods, feeds, industrial feedstocks and fibre products.

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Coproduits et quasi-coproduits de l'éthanol carburant obtenu par fermentation des céréales

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Sommaire exécutif

La technologie utilisée pour convertir la matière végétale en alcool est à la fois ancienne et bien au point. Il y a cependant matière à amélioration, tant dans la technologie des procédés que dans l'utilisation des coproduits. Une exploitation plus intensive des sous-produits se traduirait par des gains d'efficacité et de rentabilité pour les producteurs d'éthanol et les agriculteurs. La création de marchés pour les coproduits et les quasi-coproduits de l'éthanol assurera l'avenir de l'industrie de l'éthanol.

Étant donné que l'utilisation de coproduits se veut dans bien des cas une solution pour l'élimination des déchets, le potentiel des coproduits n'est pas toujours exploité pleinement. La plupart du temps, les sous-produits de l'éthanol carburant sont utilisés dans l'industrie des aliments pour animaux. Si l'on peut développer des produits pour d'autres marchés, le potentiel de rentabilité s'en trouve accru. On met actuellement au point de nouveaux procédés de biotechnologie qui permettront de produire de nouvelles matières premières pour les industries alimentaire, cosmétique et pharmaceutique.

La recherche biotechnologique est très avancée au Canada. Toutefois, certains projets de recherche prometteurs n'ont pas trouvé de débouché commercial parce que les travaux ont pris fin au banc d'essai. Dans d'autres cas, il y a eu dédoublement de recherches. On a créé une base de données de référence pour aider les chercheurs à se familiariser avec les travaux déjà effectués dans le domaine de l'utilisation des sous-produits de la fermentation des céréales pour la production d'éthanol carburant. La base de données en question est accessible sur Internet à l'adresse http://res2.agr.ca/research-recherche/cfar/coproduc.html.

Le présent rapport porte sur les quatre petites céréales principales (maïs, blé, avoine et orge). Un sommaire des ouvrages scientifiques et techniques contemporains sur les coproduits et les quasi-coproduits de l'éthanol carburant issu de ces céréales a été préparé. Nous avons laissé de côté la question de l'utilisation dans l'alimentation animale pour nous concentrer sur les usages à plus forte valeur ajoutée.

Le type de coproduits dépend de l'aliment pour animaux et de la technologie de transformation utilisés. L'amidon est consommé au cours de la fermentation pour produire de l'éthanol, tandis que les principaux sous-produits sont des protéines et des fibres. Les protéines ont été analysées sous forme de concentré pour l'alimentation humaine et de matière industrielle. Les fibres céréalières ont suscité beaucoup d'intérêt ces dernières années, car on leur associe certains effets bénéfiques pour la santé.

D'autres constituants minoritaires ont également été étudiés aux fins de l'extraction avant ou après la fermentation. En outre, nous avons évalué la possibilité d'utiliser certains sous-produits tels la vinasse d'éthanol comme milieu de culture microbienne pour la production de composés à valeur ajoutée, y compris d'acides et de pigments organiques.

Nous avons étudié les marchés actuels des dérivés de céréales pour évaluer la demande de coproduits et de quasi-coproduits de l'éthanol. En connaissant mieux la façon dont les constituants des céréales sont maintenant utilisés, le secteur et les chercheurs peuvent concentrer leurs efforts sur les intérêts commerciaux potentiels.

Les dérivés des céréales sont utilisés dans les industries alimentaire, cosmétique et pharmaceutique. Certains constituants sont reconnus pour avoir des effets importants sur la santé lorsque incorporés dans le régime alimentaire de personnes souffrant notamment de cardiopathie, de diabète et de

cancer. Le développement des marchés des aliments fonctionnels ou nutraceutiques pourrait jouer un rôle important dans le futur.

Différents constituants des petites céréales entrent dans la fabrication de certains cosmétiques, produits de toilette et produits pharmaceutiques. Il s'agit des protéines et de leurs motifs structuraux, des acides aminés, des corps gras, des fibres, des vitamines et d'autres constituants mineurs. S'il est possible de mettre au point des méthodes d'extraction rentables, certains dérivés pourront éventuellement remplacer les composés synthétiques utilisés aujourd'hui. Une telle substitution serait avantageuse pour le marché des produits dits « verts » ou biologiques, qui a connu une forte croissance ces dernières années. Ce sont les créneaux commerciaux ou les marchés nouveaux, plutôt que les marchés déjà très compétitifs et bien approvisionnés, qui offrent les meilleures possibilités de développement.

Les nouveaux établissements de production d'éthanol au Canada savent très bien qu'il est nécessaire d'exploiter systématiquement tous les constituants des céréales destinées à l'alimentation des animaux. L'éthanol utilisé comme carburant et (ou) à des fins industrielles demeurera bien sûr un produit important; toutefois, les producteurs d'éthanol carburant cherchent à créer un certain nombre de catégories de produits pour diversifier leur potentiel de profit. Des techniques de prétraitement pour la séparation de certaines fractions des céréales avant la fermentation sont actuellement à l'étude.

Nous avons dressé un sommaire des travaux de recherche du secteur, des universités et des gouvernements. À cette fin, nous avons puisé des renseignements dans diverses sources, y compris dans des bases de données électroniques (Inventaire de la recherche agroalimentaire au Canada - IRAC; Current Research Information System - CRIS, États-Unis; Australian Rural Research in Progress - ARRIP; Inventaire permanent des projets de recherche agricole - AGREP, Union européenne; Crop Association Sponsored Research Archive - CASRA, États-Unis), ainsi que dans des comptes rendus de conférences, des publications courantes et des communications avec les établissements de recherche du secteur, des universités et des gouvernements. Nous avons préparé des articles et les avons fait paraître dans deux publications, Biomass & Bioenergy Innovations (anciennement Bioenergy West) et The Energy Independent, pour promouvoir le projet et demander de la documentation.

Les États-Unis, dont l'industrie de l'éthanol est beaucoup plus vaste que celle du Canada, effectuent actuellement des travaux importants dans le domaine de la recherche sur les coproduits. Ces travaux portent principalement sur le maïs, qui représente de loin la plus importante matière première pour la production d'éthanol aux États-Unis. On étudie de nombreux aspects de la production d'éthanol à partir de maïs qui on trait à la production de coproduits, notamment la modification génétique de cultivars de maïs, la technologie des procédés, les méthodes d'extraction, les nouvelles applications industrielles et la surtransformation à valeur ajoutée de sous-produits comme les fibres de maïs.

Au Canada, les travaux de recherche qui portent directement sur les coproduits de l'éthanol obtenu par fermentation de petites céréales sont négligeables. Toutefois, la recherche sur la transformation à valeur ajoutée et l'utilisation des constituants mineurs des céréales, surtout ceux du blé, de l'avoine et de l'orge, suscite beaucoup d'intérêt. Une usine d'éthanol peut appliquer certaines des découvertes faites en laboratoire pour créer des catégories de coproduits et il existe des plans pour le faire dans un avenir prochain. À la lumière des études actuelles, ce sont les fractions de fibres insolubles et solubles qui semblent offrir le plus de possibilités commerciales.

L'industrie canadienne de l'éthanol carburant est à la croisée des chemins. Son développement permettra d'améliorer la viabilité des collectivités rurales, d'ouvrir des possibilités économiques dans le secteur agroalimentaire et de préserver l'environnement. Cependant, la production d'éthanol carburant ne peut se rentabiliser à elle seule. Il importe d'aménager des bioraffineries de céréales qui créeront, en plus de l'éthanol, d'autres catégories de coproduits comme des aliments pour la consommation humaine et animale, des matières premières industrielles et des produits fibreux.

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Chapter 1. Review of the Literature

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1. BACKGROUND INFORMATION

1.1 Introduction

Renewable sources of industrial feedstocks will increase in their attractiveness in the future as non-renewable resources, primarily fossil fuels, are depleted (Lipinsky, 1981a,b; Simmonds and Orth, 1973). By the year 2000, it has been projected that over half of the world's oil will have been consumed, generating shortages and price hikes (Giampietro and Pimental, 1990). Our ability to produce agricultural commodities for food has surpassed our present needs. Forward (1994) reported that it has been predicted that by 2010, one half of agricultural output may be directed towards non-food usage.

Almost all of the chemicals that are currently in use can be generated from cereal crops using microbes or enzymes (Linko and Linko, 1981). Use of biomass as raw material can broaden the options of the chemical industry giving it more flexibility and a broader range of products (Polman, 1994). What will first be necessary, however, is a change in the mindset of the chemistry field, which has not always regarded environmental chemistry as being wholly valid (Amato, 1993).

The use of plant material, or biomass, to produce industrial chemicals has a very long history. Many of the so-called "novel" uses that are being proposed, rely on prior discoveries (Tsao et al., 1987; Vijaikishore and Karanth, 1986). The use of plant-based material in industrial chemicals decreased from approximately 35% in 1925 to less than 16% in 1989 (Forward, 1994). Recent exploitation of phytochemicals has diminished simply because of the relative ease and economy of producing identical commodities from petrochemicals. The increased use of agricultural materials and biological processes to produce industrial commodities is inescapable and as a result has a high degree of growth potential (Forward, 1994; Olivier, 1980).

In the past few years, consumers have become increasingly concerned about the environment and resource depletion. Consequently, demand for bio-based products has increased (Lee et al., 1994; Narayan, 1994). Chemicals produced from biomass are often more environmentally friendly than those produced from petrochemicals, in that they generally require production processes with less intense conditions of temperature and pressure (Olivier, 1980). They are often highly specific and seldom result in large quantities of unknown or toxic byproducts. Ethanol fuel, in particular, is seen as being more environmentally friendly than fossil fuels, that have been linked to urban pollution and global warming.

1.2 The Fuel Ethanol Industry

1.2.1 Introduction

Ethanol has been a key industrial chemical for many years. In addition to being used as a motor fuel since internal combustion engines were invented, it is the raw material for hundreds of chemicals used in foods, medicines and pharmaceuticals (Dale, 1991). The attractiveness of ethanol use in motor fuel increased in the 1970's when a major international petroleum crisis occurred. Ethanol was

seen as an extender or even a replacement for gasoline and more than a hundred ethanol plants were constructed in the U.S. during the ensuing decade. By mid-1988, only 51 were still in operation and only 28 encompassed the entire process from intact grain to ethanol.

1.2.2 Fuel Ethanol in Canada

The fuel ethanol industry began in Canada in 1980 (Boland, 1995). In 1986 only one fuel ethanol plant was operational (McCurdy, 1986). The Mohawk Oil Inc. plant in Minnedosa, Manitoba was producing approximately 4 million litres of ethanol per annum using a feedstock of 20% barley and 80% corn. They have also used 100% barley or wheat during different periods. Ten years later, Mohawk is still in operation and has recently announced expansion plans for their plant, in order to produce a patented fibre/protein coproduct for the food industry, called Fibrotein.

Commercial Alcohols Inc., of Tiverton, Ontario has supplied gas stations in Ontario with ethanol since 1992. They have a 20 million litre/annum plant which produces industrial and fuel ethanol using dry grain grinding techniques (Fairlie et al., 1994).

A 10 million litre/year plant using wheat as a feedstock is located in Lanigan, Saskatchewan (Cemcorp, 1992). The Pound-Maker Agventures Ltd. plant, a joint venture with Mohawk Oil, has a cattle feedlot on site for disposal of byproducts.

The St. Lawrence Starch Co. of Port Credit, Ontario built a 20 million litre/annum plant in 1978 to produce ethanol, mainly for industrial and beverage use (Wayman and Parekh, 1990). At the height of their production, they were processing 750 tonnes of corn per day. However, the plant was closed in 1990 (Cemcorp, 1992).

Boland (1995) reported that Canadian demand for fuel ethanol has surpassed production. There are over 600 retailers selling gasoline containing ethanol, more than half of which are concentrated in Ontario and Quebec. At the time Boland's article was written, a number of new ethanol production plants were being proposed for Ontario including a 150 million litre plant by Commercial Alcohols Inc. at Chatham, Ontario, a 52 million litre plant by Seaway Valley Farmers Energy Cooperative at Cornwall, Ontario and a 38 million litre plant by Metalore Resources at Simcoe, Ontario.

Plants ranging from 10-150 million litres are under discussion in other parts of Canada, but are farther from commencement of production. Since Boland's article, the Commercial Alcohol plant has been delayed for a number of reasons including a significant rise in corn prices, emphasizing the strong link between fuel alcohol profitability and feedstock cost, and thus the need for high-value coproducts.

The use of grain for ethanol production opens up a large new market for grain producers. By the year 2000, gasoline consumption in Canada has been projected at 34.5 billion litres (Barclay, 1992). If ethanol was added to motor fuel at a concentration of 8%, this represents a total of 280 million litres of ethanol. To produce this amount of ethanol, approximately 750 kilotonnes of grain would be required. Forward (1994) reported that the Canadian government's ethanol initiative should result in demand for grain to rise to as much as one million tonnes per year which would realize $50 million per year in net farm income.

Benefits of developing a fuel ethanol industry in Canada include diversification of the agri-food industry, economic development, sustainability of the rural communities and protection of the

environment (ICAST, 1994).

1.2.3 Fuel Ethanol in the United States

In the United States, the ethanol industry is dominated by Archer Daniels Midland Corporation that lays claim to more than half the national output (Lee et al., 1994). In 1992, 16 of the 32 ethanol plants in operation in the U.S. accounted for 90% of the total output. The industry is continuing to expand, however, with new players, such as Cargill, entering the market. In 1995, 110 million gallons of production were reportedly under construction, almost half of this in Minnesota (The Energy Independent, 1995).

1.3 Coproducts of Fuel Ethanol Production

1.3.1 Introduction

The development of the fuel ethanol industry has been constrained by economics, with the exception of a brief period during the Second World War. Without subsidies or tax credits, the only way the industry can survive is by the development of high-value coproduct streams that will offset the high cost of the feedstock (often 50% of production costs) and of the production process (Chang et al., 1995; ICAST, 1994; Beaulieu and Goodyear, 1985). Opportunities for production of enzymes, inhibitors, binding agents, etc., that are of high quality and purity, for use in medicine, pharmaceuticals and biotechnology are growing (Murray et al., 1987). These materials often sell for as much as $10,000 per gram. In Canada, the coproduct market is still greatly underdeveloped (ICAST, 1994).

Traditionally, the animal feed industry has provided an outlet for ethanol byproducts. However, these markets are now becoming saturated and new uses will be needed if profits are to be satisfactory (Hojilla-Evangelista et al., 1992c). Products entering the human and pet food markets are generally of higher value than those destined for the feed industry, and therefore some additional investment is cost-effective, in order to further process and upgrade a product (Gras and Simmonds, 1980). If ethanol production coproducts are to be utilized for human food, they will be required to satisfy government food regulations, which will add to capital costs (Anonymous, 1981).

The type of coproducts that are produced depends on a number of factors, including conversion technology, feedstock and milling process (Turhollow and Heady, 1986). New technologies now under development, including cell immobilization, extractive fermentation, very high gravity fermentation, cellulose conversion, membrane technology, etc., will affect the amounts and types of byproducts produced (Cemcorp, 1992; Sroka and Rzedowski, 1991; Amin et al., 1983). While it is difficult to foresee which coproducts will become the most successful, profits associated with the sale of a number of materials may become as important to the industry as innovations which increase the efficiency of the ethanol production process (Rendleman and Hohmann, 1993).

Although it has been suggested that coproduct revenue can account for up to 40% of the income of an ethanol plant (Chang et al., 1995), it is difficult to put a value on novel coproducts because many that have been proposed do not yet have established markets (Spelman, 1994). In addition, if ethanol production were to grow significantly, producing massive amounts of byproducts, the value of coproducts may diminish because of oversupply. Lee and associates (1994) proposed that in the future, commodity and merchandising innovations will cause the byproduct market to change dramatically as niche products and demand for these new products is created.

Development of high-value coproduct markets will necessitate intensive technological and market development in the areas of cosmetics, pharmaceuticals, polymers, and carbon dioxide (ICAST, 1994). One of the areas of great interest at present, is that of nutraceuticals or functional foods, i.e. foods or food components that have been shown to have either health or medical advantages (DeFelice, 1995). These may include dietary supplements, isolated nutrients or processed foods, and many can be derived from cereals. A number of byproducts from the fuel ethanol industry may have potential for application in this area. Since this represents a huge potential market, still in its initial development, researchers should not lose sight of this opportunity.

The consumer has always purchased particular foods or diet supplements because of information they have received about potential health benefits. In recent years, researchers have been able to isolate some of the components in particular foods which are active agents (e.g. tocotrienols, ß-carotene, ß-glucan, lignins, phytoestrogens, phenolic acids, plant sterols, oryzanol, and various antinutrients (i.e. phytic acid, tannins and enzyme inhibitors) (Thompson, 1992). The American National Cancer Institute has identified both wheat and oats as meriting further study because of the components they possess which have been linked to disease control (Wrick, 1993).

One of the constraints on the development of functional foods or nutraceuticals, is the regulatory procedure that must be followed. In order for a health claim to be made for a particular food or plant derivative, it must first undergo extensive testing as a drug, which is a lengthy and costly procedure.

1.3.2 Biorefinery Concept

The concept of a biorefinery that produces a stream of products using biomass as a feedstock is by no means new (Broder and Barrier, 1988). Each plant could be designed to take advantage of differences due to location, including feedstocks, markets, transportation costs, regulations and subsidies. A biorefinery for grain could produce a number of value-added products, in addition to ethanol. In fact, ethanol may become the coproduct in some instances, while a value-added commodity such as a fibre or protein product may be the major source of income for the plant (ICAST, 1994).

Depending on the biomass utilized, products such as CO2, glycerol, lipids, oils, citric acid, lactic acid, acetic acid, methanol, isopropanol, xantham gum, protein polymers, pullulan, etc. could be produced for use in the food, cosmetic and pharmaceutical industries (Wyman and Goodman, 1993a,b; Maisch, 1987). New products, which meet specific market needs not already met by petrochemical materials, should receive primary attention at this point; e.g. new adhesives, biodegradable plastics, degradable surfactants, specific polymers and enzymes. Product development to fill particular niches will be crucial to success. Biotechnology will undoubtedly play a significant role in coproduct exploitation.

1.3.3 Protein

The recovery of protein from fuel ethanol production byproducts has been reviewed by a number of researchers including McCurdy (1986) and Dale (1983). Protein is seen as the major byproduct of ethanol production, because of its higher value relative to other possible derivatives. Protein is worth approximately three times that of an identical weight of sugar and it has an established market in the feed industry (Broder and Barrier, 1988). Protein can be extracted from the production stream either as a pre-fermentation or a post-fermentation step. As a pre-treatment, wet milling or dry

processing/air classification can be employed (McCurdy, 1986). Generally speaking, if coproducts such as protein are removed before hydrolysis and fermentation, they will be of higher quality. Their actual nutritional value and composition are dependent upon a number of processing factors (Satterlee et al., 1976).

1.3.4 Fibre

Dietary fibre is composed mainly of nonstarch polysaccharides and lignin (Thompson, 1992). The insoluble form of dietary fibre includes cellulose, lignin and hemicelluloses while the soluble portion consists of pectins, beta-glucans, gums and mucilages. Different grains have been found to have different proportions of the different types of fibre and this will affect whether or not they are associated with any health benefit. Barley and oats, for example, have greater concentrations of soluble fibre than do wheat and corn. Dietary fibre has been linked to control of heart disease, cancer, diabetes and obesity.

1.3.5 Carbon Dioxide

During the conversion of starch to ethanol, CO2 gas is produced. In fact, almost half of the glucose by weight is lost in this manner (Maisch, 1987). In some cases this can be sold for use in freezing foods, charging fire extinguishers, making dry ice, carbonating soft drinks, etc., to increase profitability (Dale, 1991; Keim, 1983). In all cases, sale is dependent upon having a market nearby (Scheller, 1981). The installation of recovery equipment is only cost-effective in large plants where significant volumes of CO2 are produced (McCurdy, 1986).

Current research is looking into new high value uses for CO2. It has been proposed that ethanol plants could be tied to greenhouse facilities which would use the CO2 (Cemcorp, 1992; Scheller, 1981; Hayes and Timbers, 1980). Researchers are also attempting to identify a bacterium that could convert the carbon dioxide to acetic acid (Rendleman and Hohmann, 1993).

1.3.6 Minor Components

The major cereals (wheat, corn, barley and oats) have unique chemical compositions. Compounds have been identified in each, which if potentially extracted as coproducts of fuel ethanol production, could have use in foods, pharmaceuticals, cosmetics, or the biotechnology industry. Minor component recovery will be discussed for each crop individually, later in this report.

1.4 Ethanol Production Technology

1.4.1 Introduction

The production of ethanol from grain is a continually evolving process as new discoveries that can make the process more efficient and cost-effective are made and applied. Unfortunately, incorporation of new techniques into commercial enterprises has been slow (Keim, 1983).

Generally speaking, the production of fuel ethanol comprises four main steps: (1) the treatment of the feedstock to form a sugar solution; (2) conversion of the sugar to ethanol and CO2 by yeast or bacteria; (3) distillation of the ethanol from the fermentation broth, and; (4) dewatering of the ethanol (McCurdy, 1986). The actual production of ethanol, requires only the carbohydrate portion of the grain; the other materials, including protein, fibre, oil, ash and gum, are superfluous to the

process. The ultimate goal of the ethanol industry should be complete utilization of the feedstock with maximum production of ethanol and value-added products, and minimization of waste and pollution (Finley, 1981).

An array of technologies are used to produce fuel ethanol. Discussions of the different processes are found in a number of publications including Mulligan (1993), May (1987), McCurdy (1986), Keim (1983), and Pomeranz (1973). Wayman and Parekh (1990) give detailed descriptions of ethanol production at three plants, including the St. Lawrence Starch Company. Flow diagrams for wet milling, dry milling and dry grinding processes can be found in Fairlie et al. (1994). In this report, production technologies, including the traditional methods of wet and dry milling as well as some of the more recent advances, will only be touched on briefly.

1.4.2 Wet Milling

Wet milling has been used for many years in the starch industry, and in a modernized form is being adapted for fuel ethanol production (Monenco AGRA Inc., 1993). Even though wet milling combines higher capital and energy costs with lower ethanol yields, it predominates the industry because it generates a purer starch stream and higher value coproducts (Chang et al., 1995; Rendleman and Hohmann, 1993; Kane and Reilly, 1989). Market development for coproducts has been driven by the increasingly large volumes of byproducts produced and has stimulated a substantial amount of research by industry, and at university and government laboratories (Wright, 1987). The bulk of this research has been in the area of livestock and poultry feeds.

Wet milling procedures for corn and wheat are somewhat different in that the most desirable product from corn is the carbohydrate component, while from wheat the gluten is more valuable (McCurdy, 1986). Briefly speaking, the corn wet milling process involves soaking of the grain in water and sulphur dioxide for 24-48 hours, followed by grinding. The steeping process disrupts the kernel in such a manner that the oil and protein can be removed and a starch enriched product for fermentation is produced. For wheat, the bran and germ are generally removed by dry processing in a flour mill before steeping in water.

Following isolation of the starch fraction, enzymes are added to convert the starch to glucose. Yeast, generally *Saccharamyces cerevisiae*, is added to ferment the glucose to ethanol. The ethanol is distilled from the fermentation broth leaving the stillage. The stillage can be further fractionated into thin stillage and condensed distillers solubles by removal of the solids (Hayman et al., 1995).

1.4.3 Dry Milling

The dry milling industry is somewhat smaller than the wet milling industry at present, accounting for approximately 40% of the market and showing signs of gradual shrinking (Rendleman and Hohmann, 1993). While dry milling has the advantage of being less costly than wet milling, it does not have as great a potential for the production of high-value coproducts.

Dry milling makes no attempt to fractionate the different components of the cereal grain (Mulligan, 1993; Rao, 1979). It involves grinding of the grain, followed by addition of water and heat treatment (Kane and Reilly, 1989). Enzymes are added to the slurry and the sugar which results from starch conversion is fermented to ethanol by the addition of yeast. Fuel ethanol is produced by distillation and evaporation.

Following the distillation of ethanol from the fermentation broth, a product is left which is called stillage (McCurdy, 1986). The quantity of stillage produced equals 10-15 times the amount of ethanol produced, on a volume basis (Maiorella et al., 1983). Stillage can be separated into approximately equal parts of distillers' grains and distillers' solubles (thin stillage) by centrifugation or screening. The material is then usually dried via evaporation, for use in animal feed. Distillers' dried grains (DDG), distillers' dried solubles (DDS) or a combination product, distillers' dried grains with solubles (DDGS), are produced and traditionally used as animal feed.

1.4.4 Membrane Technology

Evaporation is a costly procedure in terms of energy and expense for concentrating stillage material. In recent years, significant advances have been made in the area of membrane technology for the removal and concentration of solvents from dilute fermentation beers (Qureshi and Manderson, 1995; Köseolu et al., 1991). Following the distillation of ethanol from fermentation broth, the byproduct that remains contains yeast cells, non-fermentable sugars, polysaccharides, some acids, protein and carbon. Membranes, consisting of thin sheets of semi-porous material, have the ability to economically and effectively remove, concentrate and purify some of these components so that they may be sold as high value coproducts of the ethanol fermentation process (Hohmann, 1993; Rendleman and Hohmann, 1993). By eliminating the need to use heat to evaporate and concentrate solutions, there is less heat damage to potential products (Köseolu et al., 1991). It is possible to recycle the fermentation broth after it has been purified, reducing both the need for fresh water and the cost associated with waste disposal.

Byproducts such as lactic acid, citric acid or sorbitol could potentially be separated using membranes and sold as separate high-value coproduct streams from a fuel ethanol plant (Hohmann, 1993; Rendleman and Hohmann, 1993). However, there are several variables that still need to be addressed, including durability of the membranes, clogging caused by certain substrates, safety documentation and concern about chemical inertness and pH sensitivity.

Wu and his associates at USDA, have conducted research into the use of reverse osmosis and ultrafiltration to concentrate the stillage. Reverse osmosis takes advantage of membrane technology to separate water from ions and other dissolved substances. High pressure is used to force stillage across the membrane against osmotic pressure. Wu et al. (1983) reported that ultrafiltration followed by reverse osmosis resulted in a permeate with better quality than tap water.

1.4.5 Extractive Fermentation

A new process for the production of fuel ethanol, termed extractive fermentation, has been developed over the last decade by researchers at the Chemical Engineering Department of Queens University (Fairlie et al., 1994). In this process, ethanol is formed and recovered concurrently in the same container. Extractive fermentation can be applied to both wet and dry grinding processes and has the capacity to improve cost-effectiveness.

1.4.6 Sequential Extraction Process (SEP)

Sequential extraction, a relatively new procedure, involves simultaneous drying of ethanol from 95-99.5% and extraction of oil from the grain (Chang et al., 1995; Monenco AGRA Inc., 1993; Hojilla-Evangelista et al., 1992a,b,c; Chien et al., 1988). The protein is subsequently extracted using an

ethanol/alkali treatment and can be converted into a food-grade protein concentrate. The starch is further processed to ethanol which in turn can be used in upstream extraction procedures. The SEP process is ideal for fuel ethanol production since it allows for the recovery of high-value coproducts and provides for an economical method of dewatering the ethanol (Chang et al., 1995).

2. WHEAT

2.1 Introduction

Because of its high value as a food commodity, the use of wheat for industrial purposes has been fairly limited. Non-food use of wheat will only come about if wheat prices are low or if alternative methods of producing chemicals become too costly or difficult. During the Second World War for example, when demand for fuel and chemicals for the war effort was high, industrial use of wheat for the production of alcohol peaked because of shortages of fossil fuels and other cereal grains (Gagen, 1973).

Wheat has been identified as a feedstock of choice for fuel ethanol production, not only on the Canadian prairies but also in Ontario (Warren et al., 1994; Beaulieu and Goodyear, 1985). Wheat is currently used as a feedstock in the Pound-Maker plant in Saskatchewan and the Mohawk Oil Co. Ltd. plant in Manitoba (Boland, 1995). In Europe and in Australia, wheat is considered the primary raw material for fuel ethanol production (Swinnen et al., 1988; Hunwick, 1980). It is a more expensive feedstock than corn and accounts for 60-70% of production costs (Warren et al., 1994). However, it is not possible to grow corn in many areas of Canada. In some locations, the use of wheat may be warranted because the higher quality byproducts can fill a particular market niche (Wu et al., 1984).

Dale (1991) reported ethanol yields of 340 litres/ton or 510-710 litres/hectare/year when wheat was used. Sosulski and Sosulski (1994) reported commercial yields ranging from 342-364 litres/tonne, depending on the type of wheat used. Estimates from St. Lawrence Reactors Ltd. technology were 396 litres/tonne with a byproduct output of 266 kg/tonne (based on 90% dry matter) (Beaulieu and Goodyear, 1985). The Biostil process, a continuous fermentation process that includes recycling of the fermenter broth, has reported yields of 530 kg/ton (Swinnen et al., 1988).

The profitable production of fuel ethanol fr. n wheat is dependent on byproduct credits (McCurdy, 1986). Traditionally, large scale ethanol production has been a very important source of fibre and protein byproducts for the feed industry and profitability of ethanol production has been closely tied to byproduct prices (Swinnen et al., 1988). Recently, however, other high value alternatives not before possible, have become conceivable because of new developments in process technology (Monenco AGRA Inc., 1993). These fall into the categories of pharmaceuticals, cosmetics and the exploitation of novel characteristics of specific minor wheat components for use in food.

2.2 Composition of the Wheat Kernel

The wheat kernel is composed of the germ, endosperm and bran contributing 2.5, 83 and 14.5%, respectively, to the total weight (TWG Consulting, 1995; Fellers, 1973). Since wheat has a lower starch content than corn, it is even more important to maximize the value-added potential of the byproducts formed when wheat is used as a fermentation feedstock for fuel ethanol production (Warren et al., 1994).

Thompson (1992) referred to a number of sources to provide figures for the average chemical composition of the wheat kernel. She listed protein (14.2%), fat (2.7%), fibre (12.6%) as well as minerals, vitamins, natural phenolics and phytate.

Byproducts from wheat have a greater potential for use in human food than do those from corn. This is due to their having a higher protein and lower fat content (Wu et al., 1984). The lower fat content of wheat allows for more acceptable flavour and greater storage stability. Protein content of wheat, particularly concentration of the essential amino acid lysine, is higher than found in corn.

2.3 Ethanol Production from Wheat

The methodologies traditionally used for the fractionation of wheat into its major components is described by Fellers (1973). A number of different approaches have been utilized in the past, and the desire for certain coproducts plays a role in the choice.

Traditionally, ethanol has been produced from the whole wheat grain, resulting in a byproduct with a high pollution potential, if an alternative use for it is not available (Hunwick, 1980). To alleviate this problem, one of the key steps in the production of fuel ethanol from wheat would be to separate the protein from the starch before fermentation. Gluten, which is insoluble in water, can cause problems in subsequent processing, if it is not completely separated from the starch (Weegels et al., 1992). A number of processes for the separation of carbohydrate from protein are described by Hunwick (1980) and Fellers, (1973). Recent studies by Weegels and coworkers (1992) have confirmed the usefulness of enzymes, such as cellulase and hemicellulase, in increasing yields of gluten and starch from wheat and improving wheat processing qualities.

An integrated wheat biorefinery that can produce fuel ethanol, milled wheat products and high value coproducts has long been touted as the most viable choice for the future of the wheat industry (Delmas and Gaset, 1989; Adams, 1973). Such a plant would separate the wheat into the fibrous bran hull, the germ, starch and gluten, all before fermentation. The plant, which may contain fermentation equipment for the production of ethanol and other speciality chemicals, could take advantage in advances in cellulose fermentation and provide for wheat germ oil refining (Hunwick, 1980). Market development for the various product lines would be a prerequisite, but by fully exploiting all parts of the wheat grain, such a plant could minimize waste disposal problems and diversify its output production so that its economic survival was more assured (Adams, 1973).

Production efficiency of ethanol processing is reduced when the whole grain, including germ, bran and outer endosperm proteins that do not contribute to ethanol yield, are carried through the fermentation (Sosulski and Sosulski, 1994; Simmonds et al., 1981). Fractionation of the fermentable from the non-fermentable portion of the grain, has the potential to increase the efficiency of the process and to produce higher quality byproducts. Dexter et al. (1994) found that preprocessing durum wheat, for example, had advantages in spaghetti production in terms of colour and general appearance.

Sosulski and Sosulski (1994) preprocessed wheat grains before fermentation to ethanol. They used hard red spring, soft white winter and a cultivar of the new Canadian prairie spring wheat, which is a high yielding type with 5% more starch and less protein. They employed a dry milling technique involving smooth and corrugated rollers and screening in progressive stages. The bran fraction removed represented 22.0-24.2% of the original grain weight but in addition to fibre, contained 7.5-

9.2% of the grain starch, which would then be unavailable for conversion to ethanol. However, preprocessing the grain would have an economic advantage for the plant since the fibre coproduct which results has a higher inherent value than either the ethanol or the dried stillage.

Post-fermentation byproducts from Sosulski and Sosulski's (1994) process included CO2 and dried stillage, representing 28.5-30.4% and 40-43% of the total byproducts, respectively. The dried stillage had a protein content ranging from 31.5-35.1%, total digestible fibre (TDF) from 24.0-27.4% and fat from 5.3-6.3%. The remainder of the dried stillage was comprised of ash, phytate, glycerol, organic acids and residual starch.

Research into very high gravity fermentation (VHG) of wheat to produce ethanol is being done at the University of Saskatchewan (Jones and Ingledew, 1994a,b; Thomas et al., 1993a,b). The VHG process involves removal of the bran before fermentation which, again, opens the door for value-added coproduct production.

Collins and Paton (1991,1992) patented a process for fractionation of cereal grains, particularly wheat and oats. The process involved wet milling grain that had been soaked in aqueous alcohol. Using screens of different mesh sizes, they obtained four wheat fractions - three flours with different protein contents ranging from approximately 6-22% (dry basis) and a bran fraction which contained 16% protein. Collins and Paton suggested that anionic or cationic exchange resins could be utilized to treat the ethanolic waste solutions to further extract value-added components. They were able to detect the presence of a number of phenolic acids, fatty acids, phosphatides, organic acids, amino acids and uronic acids.

2.4 Potential Coproducts of Ethanol Production from Wheat

2.4.1 Protein

Wheat proteins have been found to have many applications both for food and for industrial uses. Gagen (1973) reported that wheat protein has been utilized in construction materials, plastics, chewing gums, pharmaceuticals, coatings and sausage casings. In 1980, non-food utilization of wheat protein was practically nil (Gras and Simmonds, 1980). However, Gras and Simmonds predicted that we may see a move back to the utilization of wheat protein in certain applications where a hard, highly cross-linked material is required for some industrial purpose.

Wheat has an approximate protein content of 10-13% by weight, 80-90% of which is made up of gluten (Weegels et al., 1992; Köseolu et al., 1991; Krull and Inglett, 1971). First isolated over 200 years ago, gluten remains an important additive in bakery and breakfast foods and in processed meat and fish products (Satterlee, 1981). A great deal of research has been conducted to maximize its usefulness (Curioni et al., 1995; Anonymous, 1992; Lawhon, 1987; Rennes and Lippuner, 1978; Walon, 1978; Kerkkonen et al., 1975; Kissell et al., 1975; Rao and Gerrish, 1973; Krull and Inglett, 1971; Fellers et al., 1966).

Wheat gluten can be separated from the flour to produce a light tan-coloured powder containing 76-80% protein (Rawlinson, 1975). Besides its high protein content, gluten has a bland flavour, the ability to absorb more than two times its weight in water and elastic properties that make it valuable in baking, the production of breakfast cereals, and in meat products. The unique cohesive and elastic characteristics that gluten exihibits are generated by its makeup of two main proteins, gliadin (39.1%) that is soluble in 70% ethanol and glutelin (35.1%) that is not (Köseolu et al., 1991; Krull

and Inglett, 1971). Gluten and its derivatives have considerable foam stabilizing activity that is beneficial in certain applications (Gras and Simmonds, 1980). De-aminated gluten preparations have been used in fat-reduced dairy spreads as emulsion stabilizers.

Gluten can also be phosphorulated to enhance its water binding capacity or succinulated to become more soluble. Köseolu and coauthors (1991) reported that gluten can be used as an alternative to casein, a more costly protein produced from milk. It can also be used to supplement soy or other oilseed protein concentrates to produce a better balance of amino acids, and hence a more nutritious product.

The use of wheat gluten in non-food industries is dependent on finding means of modification that would allow it to be suitable for production of fibre, film, adhesives or other products (Krull and Inglett, 1971). Research has been conducted into a number of these areas. However, performance must match that of synthetic products or products from other plant sources, if commercialization is going to be possible.

Simmonds et al. (1981) and Batey et al. (1982) described a modified wet milling process whereby the wheat kernel was fractionated into bran, germ, starch and protein. The bran and germ were removed from the kernel by alkaline treatment and subsequent screening. The starch was removed from the screened liquor by a centrifugation step, leaving the dispersed protein. The protein was isolated by pH alteration, followed by further centrifugation. The resultant material, containing 95% protein, was dried to form a light, tan-coloured powder. This protein concentrate was different from gluten in that it was more extensible and was easier to blend with other materials in extrusion cooking. The authors felt that a water-soluble derivative of the byproduct, formed by alkaline deamination, would have a number of possible industrial uses and could tap additional markets, as well as those already in place for wheat gluten.

The highest concentrations (70-75%) and best quality proteins are found in the bran layer of the wheat (Research Association of British Flour Millers in TWG Consulting Inc., 1995). Isolation of the protein from the bran has proved a challenge to numerous researchers and a number of techniques have been developed. A protein concentrate from hard red spring wheat bran was produced for human consumption by Hansmeyer and coworkers (1976). By washing, spray drying and screening, they produced a high protein flour that they used successfully to fortify bread and pasta.

2.4.2 Fibre

A recent study by TWG Consulting Inc. (1995) for Agriculture and Agri-Food Canada gave a detailed report on the development of coproducts from wheat bran. The potential for removing the bran from the wheat before processing has been contemplated for many years (Dexter et al., 1994). Recently, success has been achieved with development of a number of processes using adapted rice polishing techniques (Tkac, 1992; Wellman, 1992).

Wheat bran is composed of seven layers, which from the inner to the outer include aleurone cells, nucellar tissue, seed coat, tube cells, cross cells, hydrodermis and epidermis (TWG Consulting, 1995). Tkac and Timm Enterprises Ltd., a Canadian company, is able to separate the bran and endosperm components of the wheat grain and further segregate the bran layers, thereby opening the door for the development of a number of value-added products. These products have physiochemical properties and possible nutritional attributes that are uniquely different from wheat products that have been available up to the present time.

In the Tkac and Timm process, the wheat kernel is fractionated into five product streams, including starch, wheat germ and three bran components. Seventy-five percent of the bran is sequentially stripped from the kernel using patented friction and abrasion techniques involving modified rice polishers. The bran layers have much lower starch contents than found in commercial wheat bran and can be fractionated by capitalizing on their unique physical and chemical properties then recombined to produce different products. At this point in the production stream, the preprocessed wheat can go directly to ethanol fermentation or be further processed to remove the germ and the remaining 25% of the bran located in the crease of the grain. Because the process is more efficient at removing the bran, the purity of the starch destined for ethanol production is higher, the ethanol concentration in the beer is enhanced and lower amounts of distillers' dried grains (DDG) are produced (Tkac and Timm Enterprises Ltd., 1995; Sosulski and Sosulski, 1994).

One of the strengths of the Tkac and Timm process is that it removes the bran prior to the fermentation process. The byproducts therefore have not undergone any chemical alteration caused by heat or moisture and are closer to their original form than byproducts that are extracted after the fermentation and distillation steps. Byproducts resulting from the Tkac process are being produced and used in Europe in the feed industry. A fibre product, PrimAfibre, is being marketed for human consumption. Promotional material indicates that it has double the fibre content of wheat bran and only 20% of the phytate. It has a high water absorption capacity and can be used in a variety of cereal-based foods.

Fibre, especially water-soluble fibre, has been shown to have health benefits when included in human diets (TWG Consulting Inc., 1995; Alberts et al., 1990; Walker, 1974). Connections have been made between dietary fibre and certain types of cancer, heart disease, and diabetes. Wheat bran sales soared in the mid-80's when cancer prevention was linked to the consumption of fibre (Wrick, 1993). The role of bran in cancer prevention has not been wholly characterized and continuing research seems to suggest that no single cereal component or family of components is entirely responsible. Wheat bran may also have an effect on estrogen metabolism, as found by Rose and coworkers in 1991. Similar effects were not found for corn or oat bran (Wrick, 1993).

Because of the potential health benefits associated with increasing fibre consumption, a great deal of interest has been expressed in research to increase the use of cereal bran in baked goods, including cookies, bread, cakes and crackers (Andersson et al., 1981). Andersson and associates used extrusion cooking to develop a high fibre crisp bread product using 30% bran, 60% secondary starch and 10% gluten. Extrusion cooking requires less energy, space and capital investment than conventional cooking and involves high temperature and pressure treatments over a short time. They produced a crispbread with acceptable flavour, texture and structure. However, how the process affects the nutritional quality of components such as fats, proteins and carbohydrates is still not well known.

Gagen (1973) reported that wheat bran has also been studied for use as a medium for the production of penicillin, a source of antibiotic agents and for use in protective films.

Spent yeast cells from wheat fermentation have been investigated as a source of glucan. Neto and Diaz (1994) indicated that yeast cells presently have a low market value and are generally dehydrated and sold or disposed of as waste. There is value-added potential in spent yeast, however, as yeast extract, protein isolate, nucleic acid concentrate or as crude glucan that could be used as a thickening agent in food or cosmetics. The thickening strength of crude glucan is not as great as found for some

traditional materials used for this purpose, but was effective if used in high enough concentration. Used in low-calorie foods, glucan gives a fat-like mouth feel.

2.4.3 Germ

Wheat germ is particularly attractive for use in food products because it contains high concentrations of protein and minerals, a number of vitamins including Vitamin E, and its oil has a greater percentage of unsaturated fatty acids than do animal fats (TWG Consulting Inc., 1995; Tsen, 1980). It has a protein content of 26-28% and a lipid content of 20%. Commercial wheat germ includes small amounts of bran and endosperm

The use of wheat germ in bakery products was reviewed by Tsen in 1980. Wheat germ can be used to fortify baked products such as bread, cakes and cookies, but baking quality will be affected and processes must be modified if an acceptable commodity is to be produced. Wheat germ is particularly attractive as a protein supplement because it contains adequate amounts of lysine.

2.4.4 Distillers' Dried Grains (DDG), Distillers' Dried Solubles (DDS) and Distillers' Dried Grains with Solubles (DDGS)

DDG, DDS AND DDGS which result as byproducts from the dry milling process are generally sold as animal feed. However, they do contain a high concentration of dietary fibre and protein and have potential for increased value if used as a flour supplement in baked goods.

A great deal of research was done on DDGS at the Institute for Food Science and Technology at the University of Washington in the late 1980's, the premise being that fuel ethanol production from wheat could not be profitable unless a high value use, such as human food, was found. Rasco and associates (1987a) determined the chemical composition of DDGS from soft white wheat and hard red wheat. Protein in the DDGS was concentrated 2.4-2.9 times and ranged from 19.6-38.4%. The amino acid profile did not change during the ethanol production process and lysine remained the limiting amino acid (Dong et al., 1987).

Crude fibre in the DDGS was concentrated 2.6-3.8 times and ranged from 6.8-8.0% (Rasco et al., 1987a). Total dietary fibre comprised approximately one third of the weight of the DDGS (San Buenaventura et al., 1987). On a dry weight basis, DDGS from soft white winter wheat and hard red winter wheat was composed of 20-40% neutral detergent fibre, 10-14% acid detergent fibre, 8-9% crude fibre, and 3-4% lignin (Dong and Rasco, 1987).

Rasco and coworkers (1987b) substituted 30% of the all-purpose flour in white bread, whole wheat bread, chocolate chip cookies and banana bread with DDGS. The cookies and banana bread received ratings from sensory panels that were as good as those for the controls containing no DDGS. The breads were rated acceptable to good. Nutritionally, dietary fibre and protein were increased in the DDGS products by 140-500% and 130-150%, respectively. When DDGS was added to fish batter, replacing 25% of the all-purpose flour, the product was acceptable to panelists despite the darker colour, and in fact a number of panelists preferred the yeasty flavour which resulted (Rasco et al. 1987c).

In 1989, Rasco, McBurney and Edmonds, patented a human food product which they produced from DDGS. The primary drawbacks to the utilization of DDGS in human food are smell and taste. A number of researchers had attempted to alleviate these problems with little success until Rasco and

associates developed their process which involves adjusting the pH of the stillage using organic and inorganic acids and neutralization with selected hydroxides or oxides before drying to a moisture content of 5-10%. The drying temperature must be kept low so as not to adversely affect colour or flavour. The product could be used in baked goods such as brownies, cookies, pasta, yeast breads and quick breads at concentrations ranging from 10-50%, depending on the product.

Kim and coworkers (1989) looked at the use of dried distiller grains (DDG) in extruded snack products. Extrusion cooking involves the cooking of moist starchy or proteinaceous materials in tubes by a combination of pressure, heat and mechanical shearing (Hauck, 1980). The extrudate is generally formed by passing it through different shapes of openings in the final die. Wheat DDG was found by Kim and coworkers (1989) to be one of the more successful materials for extrusion when compared to corn, oats, barley, rye and sorghum. Wheat DDG contained 26% protein, 18% fibre and 11% fat.

Wu and coworkers (1984) looked at the byproducts formed when hard and soft wheats and their flours were fermented to produce ethanol. In addition to the energy costs associated with drying stillage to concentrate the solubles, they found problems with denaturation of the protein that could interfere with use in food products. Wu (1987) used reverse osmosis and ultrafiltration to fractionate the stillage into different components, leaving a permeate that could be recycled through the system. The process was less costly than evaporation and produced a product that had potential for incorporation into foods such as baked goods. Concentrated wheat distillers' grains from the fermentation of wheat flour may have use in products such as baby food, where a low fibre content is desirable (Wu et al., 1984).

2.4.5 Minor Components

2.4.5.1 *Phytate and Phytate Derivatives*

Phytate and phytate derivatives (e.g. phytic acid) are found in both wheat germ and bran (Murray et al., 1987). Thompson (1992) reported a level of 2.9 mg/g in whole wheat grains. Phytic acid has potential use in a number of medical applications. It has been proposed as an imaging agent for organ scintigraphy and as a contrasting agent for X-rays taken using barium sulfate. Because of its ability to chelate cations, it may provide an antidote to lead poisoning and reduce calcium deposition. Phytate and derived compounds may have some use in the treatment of heart disease, certain types of cancer and diabetes (TWG Consulting Inc., 1995; Thompson, 1992). Use in dentistry as a cavity prevention tool has also been suggested (Murray et al., 1987).

Because it is an antioxidant in oils, phytate may be used in food preservation (Graf, 1983). It may have application in toxic waste disposal and as an anticorrosive agent in paints and lubricant greases. At present, phytic acid is available commercially. It is not, however, of high value, and further research into uses and market development is required.

2.4.5.2 *Enzymes, Enzyme Inhibitors and Mycotoxins*

Enzymes represent a rapidly growing commercial area. A variety of enzymes with documented uses are found in wheat and can be extracted (Murray et al., 1987). Carboxypeptidase and phytase are present in wheat bran. Carboxypeptidase has demonstrated use in amino acid sequencing of proteins while phytase can be used to hydrolyze phytic acid. Acid phosphatase, lipase, sucrose phosphate synthetase and sucrose synthetase are found in wheat germ. -amylase inhibitors (water-soluble

proteins) found in wheat have potential for treating wheat to reduce sprouting during harvesting.

Enzymes and mycotoxins may also be biosynthesized indirectly from fungi growing on wheat grains (TWG Consulting Inc., 1995). They may have use in medicine and biotechnology for research processes. Development of markets for wheat-derived enzymes, enzyme inhibitors and mycotoxins should be considered.

2.4.5.3 *Cinnamates*

Certain cinnamates found in the bran of wheat have good redox properties and the ability to absorb radiation. They, therefore, could be used as the active component of sunscreens (TWG Consulting Inc., 1995). Levels found in bran, the cost of extraction and extraction methodology still need to be established. It may be possible to use some of the ethanol produced from wheat starch to extract cinnamates from wheat bran.

2.4.5.4 *Vitamin E*

Vitamin E, an antioxidant found in wheat germ, has been linked to disease prevention and is popular in certain types of cosmetics. Concentrations of 1.8-2.3 mg/100 g in whole wheat grains have been recorded (Thompson, 1992). Vitamin E may be involved in increasing the immune response in humans and in diminishing the risk of cataracts, thrombotic disease and certain types of cancer (TWG Consulting Inc., 1995). Body creams containing vitamin E are sold by a number of cosmetics firms.

2.4.5.5 *Beta-glucan*

Wheat bran contains ß-glucan, a hot water-soluble polysaccharide. ß-glucan has been linked to control of heart disease and diabetes. However, it has not been found in wheat at the high levels found in oats (Collins and Paton, 1991); wheat products have not exhibited the same hypocholesterolemic effects (Thompson, 1992) of oats. Nevertheless, consumption of whole wheat bread, rather than white bread does seem to be associated with a lower risk of heart disease.

2.4.5.6 *Glycerides*

Wheat glycerides are found in the germ and used in cosmetic preparations such as creams, lotions, lipsticks etc. as an anti-irritant (Murray et al., 1987). Since glycerides are effective in very low concentrations, demand may not be great enough to provide large market opportunities.

2.4.5.7 *Lectin*

Lectins are carbohydrate binding proteins found in the germ. They have a broad range of medical and biochemical uses based on their capacity to bind erythrocytes (Murray et al., 1987). Lectins can be bound to other molecules such as biotin or peroxidase, to increase their value. One lectin, agglutinin, can be extracted by affinity chromatography and is sold by a number of biochemical companies for as much as $1700 U.S. per gram. The magnitude of the market is not yet established, but lectins have potential use in diagnostic tests.

2.4.5.8 *Stillage Effluent*

Jones and Ingledew (1994b) reported that stillage effluent in a typical ethanol plant is 8-15 times the volume of the ethanol produced. Treatment of this material by screening, centrifugation and evaporation is expensive, so that reuse can contribute greatly to the economics of a plant.

For each kilogram of ethanol produced, 0.05-0.015 kg of yeast solids are produced (McCurdy, 1986). Jones and Ingledew (1994a,b) suggested that the spent yeast could be removed from clarified grain mashes before distillation and used as a source of nutrients in very high gravity fermentations.

3. CORN

3.1 Introduction

Corn is used as a feedstock for fuel ethanol production in Canada by Commercial Alcohols Inc. of Tiverton, Ontario and has been used at various times by Mohawk Oil in Minnedosa, Manitoba. Nearly all of the fuel ethanol produced in the United States uses corn as a feedstock, although this represents only 5% of their total corn crop or 400 million bushels (Lee et al., 1994; Turhollow and Kanhouwa, 1993). Approximately nine and a half litres of ethanol are produced per bushel of corn (Wyman and Goodman, 1993a,b). Without government subsidies or tax credits, it is generally agreed that the fuel ethanol industry could not exist, unless byproducts much more valuable than animal feed, can be developed (Turhollow and Kanhouwa, 1993; Cemcorp, 1992).

Vaughn (1995) reported that an acre of land in the U.S. can produce, on average, 115 bushels of corn which in turn can be used to produce 228 gallons of ethanol, 1437 lbs. of 21% gluten feed, 345 lbs. of 60% protein gluten meal and 173 lbs. of corn oil. Ethanol yield from corn was estimated at 380 litres/tonne by St. Lawrence Reactors Ltd. in Ontario (Beaulieu and Goodyear, 1985). Byproduct output, on a 90% dry matter basis, was estimated at 287 kg/tonne.

In 1994 there were 43 fuel ethanol plants spread throughout 21 of the American states, with a total production of more than 1.4 billion gallons (Vaughn, 1995), up from approximately 1 billion in 1993 (Wyman and Goodman, 1993a,b). This is an increase in the number of production facilities from 1992, when 32 plants were operating (Lee et al., 1994).

Eric Vaughn, the president of the Renewable Fuels Association in the United States, emphasized the importance of creating high-value markets for the byproducts of fuel ethanol production from corn. The most important variable cost factor in fuel ethanol production is the net cost of corn, which is the cost of the corn entering the plant minus the profit that can be derived from the sale of byproducts (Kane and Reilly, 1989). The price of corn tends to be much more variable than the price of coproducts which have tended to rise in recent years. The sale of coproducts from the wet milling process of producing fuel ethanol accounts for approximately 30% of revenue and more than 50% of corn feedstock cost (Lee et al., 1994). Wet milling is the process of choice in 60% of ethanol plants in the U.S.

The existence of nearly 4000 discrete uses for refined corn products were noted in 1989 (Munro, 1994). While a few products are used directly by the consuming public, most act as inputs for further processing, building layers of value-added activity.

3.2 Composition of the Corn Kernel

Corn is composed of approximately 70-75% starch, 10% protein, 4.5% oil and 10-15% other materials such as fibre and ash (Wyman and Goodman, 1993a,b; Keim, 1983). More than 75% of the protein is located in the endosperm (Reiners et al., 1973). The nutritional quality of the protein in terms of amino acid content is poor. Additionally, protein can cause problems during the milling process. Endosperm proteins are, however, nearly completely insoluble and this characteristic aids their recovery during wet milling.

3.3 Ethanol Production from Corn

3.3.1 Introduction

The fractionation of corn into its component parts has been practised for many years. The two main processes by which fractionation is achieved are wet milling, which originated in the mid-1800's, and dry milling, that was developed in the early 1900's (Rankin, 1982). Both processes are subjects of continual refinement and modification. In addition, new processes are being developed to make corn fractionation more cost effective.

3.3.2 Wet Milling

The products of corn wet milling for fuel ethanol production have been outlined by Hayman et al. (1995) and Köseolu et al. (1991). The corn kernel is presoaked and milled to produce three streams including starch, germ and fibre. The germ is extracted to produce corn oil, the most valuable coproduct of the process. The fibre portion consists of the seed pericarp and the bran, which has a composition of 70% xylose, 23% cellulose and 0.1% lignin. The starch fraction undergoes centrifugation and saccharification to produce gluten wet cake, which when dried is the second most valuable coproduct, and glucose that is fermented to produce ethanol. The ethanol is distilled leaving thin stillage, that when dewatered leaves corn condensed distillers' solubles containing 20% carbohydrate and 18% protein. The condensed distillers' solubles can be sprayed onto the corn fibre and fermented to produce corn gluten feed.

Lee and associates (1994) indicated that one of the key factors in the sustainability of the fuel ethanol industry is the development of new technology for corn wet milling. This includes, for example, techniques that would allow the cellulose in corn hulls to be converted to ethanol, resulting in an increase in ethanol yield. The coproduct stream would change significantly as a result. While less material would remain after fermentation, what was left would have a much higher protein concentration and therefore a higher value.

The need for fresh water in a corn wet milling plant is very high, reaching 1.5 m3 per ton of corn (Kollacks and Rekers, 1988). Water must be removed from the byproducts produced at various stages of the process. In order to provide an option to evaporative drying, Wu and Sexson (1985) and Wu et al. (1983) used reverse osmosis and ultrafiltration to concentrate stillage from corn grits, flour, degerminated meal and hominy feed remaining after ethanol fermentation and distillation. Most of the solids and nitrogen were recovered, leaving a final permeate that could be recycled back into the production process as water, could undergo further treatment or be discharged.

3.3.3 Dry Milling

Dry milling of corn involves breaking down the kernel to fine particles. The germ is removed by

sieving and aspiration and/or by gravity methods (Köseolu et al., 1991). Generally, prepress-solvent extraction is used to remove the oil from the germ. Milling and air classification or alkali extraction-acid precipitation processes are used to obtain protein concentrate from the defatted germ meal. Enzymes are used to convert the starch fraction to glucose and yeast is added to perform the fermentation step. From this process, 9.5-9.8 litres of ethanol, 7.3-7.7 kg of carbon dioxide and 7.7-8.2 kg of DDGS with a protein content of approximately 27%, are produced (Wyman and Goodman, 1993a,b).

3.3.4 Sequential Extraction Process

The sequential extraction process (SEP) for corn milling was developed out of a need for new low-cost methods of corn milling that could produce higher value coproducts than are currently on the market (Chang et al., 1995; Hojilla-Evangelista et al., 1992a,b,c). The two innovative features of this method are the use of ethanol in upstream steps of the process and the simultaneous extraction of corn oil and dehydration of ethanol from approximately 95%-99%. Oil yield and quality are enhanced by the SEP process since the entire kernel is extracted. There is a yield increase from 72% for conventional repress hexane extraction to 90-94% for SEP, depending on whether dent or high lysine corn is used (Hojilla-Evangelista et al., 1992b,c).

The SEP process eliminates the need for steeping in sulphur dioxide, a treatment that has been found to have negative effects on the characteristics of the protein. The SEP process generates a high quality product that can be used for food and industrial purposes. Total protein extraction ranges from 70-80%. Approximately 10% of the protein is removed in the oil extraction step. The protein, since it is soluble in ethanol, is believed to be zein. The extraction step, involving ethanol/alkali, results in recovery of about two thirds of the protein from the intact grain, depending on the type of corn used. Freeze-dried protein concentrates were produced that contained nearly 80% crude protein, compared to 60-62% in corn gluten meal. Amino acid quality was similar to that of the untreated corn and better than that of corn gluten meal. Because germ proteins are also extracted, the protein would be expected to have a higher nutritional value.

The protein produced by the SEP process was white, unlike corn germ meal which was bright yellow, and had a mild corn flavour (Hojilla-Evangelista et al., 1992b). The material had a high degree of solubility in aqueous mediums at a pH value of greater than 7. At dilute concentrations, it exhibited substantial foaming capability. It has been found to have excellent emulsifying capacity and emulsion stability, as well as good heat stability (Myers et al., 1994; Hojilla-Evangelista et al., 1992b). Because of all these characteristics, SEP protein could establish itself as a very effective material for both food and non-food uses.

After the two extraction steps, a fraction rich in fibre and starch remained. The starch was not as pure as that obtained from conventional wet milling in that it contained a higher protein concentration, but it was still a good substrate for ethanol fermentation. The fibre portion is being evaluated for use as an alternative to gum arabic (Anonymous, 1995). Chang et al (1995) concluded that before the SEP process can be commercialized, coproduct markets require development. They suggested that ethanol be considered the byproduct and that efforts should focus on the protein fraction.

3.4 Potential Coproducts of Ethanol Production from Corn

3.4.1 Protein

A great deal of research effort has gone into the development of corn protein concentrates (Shukla, 1981; Sternberg et al., 1980). A concentrate containing 90% protein has been isolated from corn gluten meal (Satterlee, 1981). The isolate was light coloured and only slightly soluble in water, but could absorb triple its weight in water and could bind its own weight in fat. For use in food, it complemented other protein sources that are rich in lysine and tryptophan, but low in methionine and cystine. For corn protein concentrates to be accepted by the food industry, they must have unique characteristics that recommend them for commercial exploitation. The ability of corn protein concentrates to complement oilseed proteins in terms of amino acid composition could be considered one of these characteristics.

At present, markets for corn protein concentrate use in food have not been developed. Chang and associates (1995) suggested that use as feed for infant animals or aquaculture may prove profitable. Zein is the only corn protein that has been developed for non-food industrial applications (Hojilla-Evangelista et al., 1992c). Between 1939 and 1967, up to 6 million pounds per year were produced (Reiners et al., 1973). The characteristics that make zein of interest for industrial application include the ability to provide a tough, glossy coating that is resistant to water, grease, scuffing and microbial attack (Reiners et al., 1973). Zein is soluble in 90% ethanol and will form a clear tough film when the solvent is evaporated. In the past, zein was utilized in the production of packaging films, linoleum tiles, coatings, ink and textile fibres, but usage diminished because of less costly petroleum based alternatives.

Currently, small quantities of zein are produced and marketed by Freeman Industries, Inc. of New York, for use as a coating on pills, nuts, candies and other foods where it forms a moisture resistant covering (Wilson, 1987; McCurdy, 1986). Narayan (1994) has been investigating the use of zein as a coating on paper or paperboard, to replace currently used polyethylene or wax coatings.

Researchers at the University of Nebraska, Lincoln, have developed techniques to co-ferment corn and whey using two different yeasts (Anonymous, 1994). Using continuous fermentation methods with columns for immobilization of yeast, they were able to combine increased ethanol yields with faster processing time, compared to conventional fermentation. A high protein byproduct was produced.

3.4.2 Fibre

Bothast (1994) suggested that value-added opportunities for use of corn fibre should be explored. Corn fibre is presently used in animal feed. However, with the growth of the fuel ethanol industry the feed market will rapidly become saturated and corn fibre, that is available at a relatively low cost, will be available for other, potentially more valuable uses. With hydrolyzation of the starch and hemicellulose fractions of corn fibre to fermentable sugars, it is possible that valuable chemicals could be produced by microbial fermentation. The unfermentable portion would be available for the feed market.

Because of the interest expressed in increasing dietary fibre, particularly in breakfast cereals and snack foods, corn bran flour was developed and test marketed in the 1980's (Alexander, 1987; Shukla, 1981). Substantial amounts of corn bran flour can be generated as a coproduct of dry milling. However, market development is a key factor and despite their advantages in terms of nutrition, colour and flavour, dry milled byproducts do not compete well with wet milled products. Corn bran has been used in non-food applications as an extender and a viscosifier for use in urea-

formaldehyde plywood adhesives (Alexander, 1987).

3.4.3 Germ

Corn germ is a byproduct of both wet and dry milling, though its protein may be somewhat altered during wet milling by the steeping process (Nielsen et al., 1973). Shukla (1981) reported that wet milled corn germ meal has a higher protein content and lower caloric value than the dry milled product. Amino acid profiles were similar. Inglett and Blessin (1979) reviewed the composition of defatted corn germ flour originating from both processes. They suggested that corn germ protein products have one of the highest potentials for use in human food. It is not as pure, flavourless or as pale as casein or soy isolates and research to improve these characteristics is necessary (Nielsen et al., 1973).

Corn germ makes up 10-20% of the total product generated when corn is dry milled (Blessin et al., 1973). Defatted corn germ flour (DCGF), developed from commercial dry milled corn germ, has been suggested for use as a protein supplement in baked goods (Nielsen et al., 1979; Tsen, 1976; Blessin et al., 1973, 1974). DCGF is high in protein, oil, minerals and vitamins; has a pleasant flavour and texture; and good hydration and emulsifying properties (Blessin et al., 1979). Addition of corn germ to cookies, bread and cakes will affect baking characteristics (Tsen, 1980). However, as long as levels are within an acceptable range, satisfactory products can be produced.

Blessin and coworkers (1973,1974) ground and screened corn germ to remove the fibre. The resultant product contained approximately 25% protein, 24% starch, 2-4% fibre and less than 0.5% fat. Use in cookies to replace 25% of the wheat flour led to increases in iron, phosphorous, potassium, magnesium, lysine and tryptophan contents. Fibre and protein were slightly elevated as well. Blessin and associates concluded that DCGF could be used in a number of foods, such as cookies, muffins and beef patties, to enhance protein and mineral contents.

Tsen (1976) used DCGF in oatmeal cookies. Taste panel evaluation indicated that cookies supplemented with DCGF in amounts as high as 48% on a wheat flour basis, had acceptable texture and taste. Indeed, cookies containing amounts up to 36% actually rated higher than the controls. Because bread forms a staple part of the diet in many countries, it is an ideal candidate for fortification. Tsen et al. (1974), reported that DCGF could be used in wheat bread at levels up to 12%. Where loaf volume is not an important factor, levels could be increased to as much as 24%. Odour and flavour were deemed acceptable.

Nielsen and associates (1979) upgraded wet milled corn germ to produce a value-added flour product that contained 30% protein, 5.9% lysine, a good balance of the other essential amino acids and a significant amount of fibre. As part of the process, ethanol was used to extract some of the oil. Nielsen and coauthors suggested that this product should be further evaluated for use in human food.

3.4.4 Gluten Meal

Steeping of corn kernels in sulphur dioxide at the beginning of the wet milling process aids the separation of starch and insoluble protein. The corn gluten meal produced is a high protein product (43-65%) and is one of the most valuable coproducts of the process (Ott and Rask, 1982, 1983; Satterlee, 1981). It contains mostly zein, but also glutelin and a small quantity of globulin (Buck et al., 1987; Wright, 1987).

Two of the problems associated with corn gluten are its unattractive flavour and odour, caused by its high unsaturated fatty acid content and its potentially harmful sulphite content (Hojilla-Evangelista et al., 1992b; Buck et al., 1987). Wu and coworkers (1994) found that treatment by either hexane-ethanol or supercritical CO2 (SC-CO2) extraction, significantly diminished the fermented flavour and made the product more acceptable for human consumption. SC-CO2 is particularly appealing as a solvent since it is non-explosive, non-toxic, easily removed from the extracted media and is not expensive. It is interesting to note that in a grain biorefinery, both ethanol and CO2 could be available for use in extraction procedures.

Buck and coworkers (1987) incorporated corn gluten meal into cookies, bread, pasta and extruded snack foods. They found that while protein efficiency was improved, flavour acceptability was diminished for all products except cookies. Texture was acceptable only for pasta. The functional characteristics of bread doughs and extruded products were affected.

Corn gluten meal can also be combined with defatted soy flour for use in food (Neuman et al., 1984). Even though zein, the main protein, contains virtually no lysine or tryptophan (Wright, 1987), corn gluten has a high sulphur amino acid content that improves the nutritional value of soy flour.

3.4.5 Oil

More than 90% of the corn oil generated in the United States originates as a byproduct of the wet milling process (Orthoefer and Sinram, 1987). Orthoefer and Sinram discussed a number of coproducts generated from the processing of corn oil. Wet gum is derived from the degumming process. It can be used with refining soapstock in animal feed or further processed to form lecithin, a potentially valuable commodity for use as an emulsifier, antioxidant, nutrient or dispersant. Vegetable oil distillate, removed from the oil during deodorization, contains a number of chemicals including tocopherols, carotenoids, flavour and colour components that can be extracted for value-added use.

3.4.6 Distillers' Dried Grains (DDG), Distillers' Dried Solubles (DDS) and Distillers' Dried Grains with Solubles (DDGS)

Walker (1980) reported that soy protein concentrates in the 1940's were in a similar position to what corn distillers' dried grains were in the 1980's, i.e. lots of potential but little application. Intensive research and marketing activity are required for DDG to become an accepted and important food commodity. Economic competitiveness, flavour, functionality and presentation to the consumer are all critical factors. Distillers' grains from different sources can vary considerably in colour, protein, fat, pH, fibre and taste and must be chosen selectively for use in human food (O'Palka, 1987).

Wu (1989) considered the effects of corn type on the residues from ethanol fermentation. In addition to dent corn that is used exclusively by the ethanol industry at present, he studied high-lysine, waxy and white corn strains. He concluded that high-lysine corn distillers' grains showed the most promise for incorporation into human food, because of their higher protein quality. White corn distillers' grains may have potential use in baking because of their light colour.

The neutral sugar contents of corn DDGS, DDG and DDS were reported by Wu (1994). He felt that determination of the carbohydrate composition of these coproducts would increase their potential for subsequent processing. DDS was found to have the highest concentration (39%) of neutral sugars.

Chemical analysis showed that glucose was in the greatest abundance, followed by glycerol. DDGS had the next highest content of neutral sugars at 38%, with glucose and xylose being predominant. DDG had the lowest content of neutral sugars at 36%, with the most prevalent types being xylose followed by arabinose.

DDGS is traditionally used for animal feed. In the United States, DDGS from the dry milling and fermenting of corn generally returns 25-50% of the original feedstock cost (Keim and Venkatasubramanian, 1989; Sauer and Compton, 1982). However, numerous researchers have suggested that if this material could be diverted to the food industry, it would have a greater monetary value and could help sustain the viability of the fuel ethanol industry.

The Cemcorp study (1992) suggested that wet distillers' grains from the dry milling process could be chemically altered to produce ingredients for the food industry. The American Xylan process can convert this material into either a dietary food supplement or an environmentally friendly barbecue briquette, both of similar value (Cemcorp, 1992). The stillage and the corn fibre also can be used to manufacture insulation and construction materials.

In addition to the yeast cells from the fermentation, DDGS contains all of the material present in the intact grain, with the exception of the starch (Scheller, 1981). Protein and fibre are concentrated three-fold in corn DDGS compared to the original grain (Rasco et al., 1987a). Levels of protein ranging from 23-35% and fibre ranging from 27-55% have been reported for DDG and DDGS (Dong and Rasco, 1987; Rasco et al., 1987a; Dawson et al., 1984; Tsen et al., 1982, 1983; Ranhotra et al., 1982). Although the protein content is fairly high, the amino acid balance is poor (Ott and Rask, 1982). Dong and co-workers (1987) studied the quality of the protein found in DDG. They found that the amino acid profile of the whole grain was not affected by the fermentation process. Lysine was the most limiting amino acid.

Distillers' grains can be used in food without restriction in the U.S. as long as the original grain used to produce ethanol is fit for human consumption and the processing plant approved for food manufacture (Anonymous, 1993). Rasco and coworkers (1987c) substituted 25% of the all-purpose flour in breading formulations with DDGS. The product had a darker colour than controls that did not contain DDGS, but was still deemed acceptable by the taste panelists.

DDGS were used in yeast and quick breads by O'Palka (1987) to replace 33 and 40% of the flour, respectively. In order for the products to be acceptable, light coloured DDGS with a pleasing smell had to be used. Sufficient quantities of baking soda were required to adjust the pH of the DDGS and additional liquid was necessary to offset the higher fibre content.

Researchers at the South Dakota State University have washed, freeze-dried, steam/pressure sterilized, oven toasted and ground DDG to produce a product suitable for use in baked goods (Anonymous, 1993). Because it contains approximately 40% fibre and 36% protein it is highly nutritious. One cup of DDG would supply the entire daily requirement (U.S.) of fibre, compared to 30 cups of corn flakes. Incorporated into human food, it would provide a significant source of dietary fibre (Dong and Rasco, 1987; San Buenventura et al., 1987).

DDG were used by Reddy and associates (1986) to supplement canned products such as stew, bean-less chili and hot dog sauce. Levels up to 2% did not significantly alter appearance, taste, mouthfeel or general acceptability. In stew, DDG acted as a thickening agent, while in bean-less chili and hot dog sauce, it replaced vegetable protein, soy or wheat flour.

DDG can be incorporated into puff-extruded products with rice, potato, wheat or corn flour doughs (Kim et al., 1989; Wampler and Gould, 1984). In terms of functionality, DDG can be used at levels ranging from 0-100% to produce extruded products (Kim et al., 1989). However, Wampler and Gould (1984) found that a mild astringent, grainy flavour could be detected at the 10% level and increased as the DDG content was augmented, becoming more unacceptable. They concluded that DDG could be used to a maximum of 20%.

Wu and coworkers (1987) considered the use of corn DDG in spaghetti. They tested this material both untreated and extracted with hexane-ethanol, at three concentrations: 5, 10 and 15%. Both products, used at the 10% level, resulted in an improvement in protein and fibre content in the spaghetti. Flavour, texture and cooking characteristics were acceptable.

The use of corn DDG in cookies and in bread was discussed by Tsen and coworkers in 1982 and 1983, respectively. DDG were found to be acceptable at levels up to 15% in bar, spice and chocolate chip cookies. Since these products normally have a darker colour than sugar cookies, the dark colour of the DDG was masked. No flavour differences were found between the supplemented and non-supplemented chocolate chip cookies. However, bar and spice cookies without the DDG were found to have significantly better flavour.

The use of distillers' dried grain flour in bread was found by Tsen and coworkers (1983) to be acceptable at a 10% level. Compared to whole wheat bread, the supplemented product had better volume, crumb grain, colour and storagability. Fibre content was lower than for whole wheat bread, but significantly higher than white bread.

One of the problems associated with utilizing corn DDG in food is poor flavour (Wu et al, 1990; Bookwalter et al., 1984). Wu and coworkers used supercritical carbon dioxide (SC-CO2) to remove the oil from distillers' grains. The resultant product received acceptable flavour scores from taste panels. Some fermentable flavour was still detectable, but even this may be masked when the product is incorporated into baked goods.

Wu and Stringfellow (1986) looked at the further processing of distillers' grains and distillers' grains with solubles in order to increase protein concentrations and reduce fibre. They found that this could be done with a simple screening process. The increase in protein content was more marked than what they had achieved previously using a more complicated dry milling procedure (Wu and Stringfellow, 1982). Selective use of screens could be used to develop a range of products with different fibre:protein ratios.

Wall and coworkers (1984) looked at the potential use of corn distillers' grains, corn distillers' grains with solubles and corn protein concentrates, produced by fermenting degermed, dehulled dry milled corn, for use as food sources in foreign aid projects. The corn products were considered as part of corn-soy-milk mixtures at levels up to 10%. The authors concluded that additional processing was needed before these products could be considered for use. Two other important factors for use of corn byproducts in food aid are taste and storagability (Bookwalter et al., 1984). Use of DDGS and protein concentrates were negated because of flavour and lysine deficiency, respectively. Distillers' grains could be used up to a concentration of 2.5% without deleterious effects. Further processing of distillers' grains by washing or defatting with a hexane-ethanol solution, led to an improvement in taste.

Wu et al. (1985) took the grits, degerminator meal, and hominy feed produced from corn dry milling and fermented each to form ethanol. The distillers' grains from the fermentation of corn grits and corn flour exhibited higher protein and lower fat and fibre concentrations than corn distillers' grains (Wu et al., 1981). Low fibre would be beneficial in the manufacture of products such as baby food where fibre is a limiting factor. Lower fat has the potential to increase storagability and may also improve flavour. The distillers' grains from the degerminator meal and hominy feed fractions were particularly rich in lysine, thus increasing their nutritional value. Wu and coworkers (1985) again emphasized the potential for developing a number of coproducts from corn fermentation to ethanol in order to fully exploit value-added opportunities.

3.4.7 Minor Components

3.4.7.1 *Stillage Effluent*

3.4.7.1.1 Introduction

Distillation of ethanol after the fermentation process leaves a primarily aqueous broth which contains organics, proteins and salts (Cheryan and Parekh, 1995; Dowd et al., 1993). In the United States, approximately 108 m3 of stillage are produced each year (Dowd et al., 1993), typically 10-15 litres of stillage per litre of ethanol produced (Maiorella et al., 1983). Most stillage enters the feed market in one form or another. However, it can also be used as a fermentation medium to produce other products, extracted to isolate minor components or recycled back into the fermentation process to provide a source of nutrients for yeast growth (Maisch, 1987).

Corn stillage from an ethanol distillery contains 7.5% solids, 2.3% protein, 1.5% ash, 0.5% sugar and a high content of vitamins on a weight basis (Maiorella et al., 1983). It has an extremely high biological oxygen demand (BOD) at 15-25,000 ppm. A 100 million litre/year plant would have a similar pollution load to a city of 1.4 million people. For this reason, treatment and byproduct recovery are very important and methodology to extract value-added material will become even more crucial as the fuel ethanol industry grows.

3.4.7.1.2 Extraction of minor components

Besides ethanol, a number of compounds are formed during the fermentation process. Dowd and coworkers (1993) used highly sensitive gas chromatography, mass spectrometry and high pressure liquid chromatography to characterize the composition of corn stillage in order to determine the potential for producing value-added products. They found that the broth contained ethanol, acetic acid, propionic acid, a mixture of higher boiling or non-volatile hydroxylated, dicarboxylic, amino and other nitrogenous acids, polyhydric alcohols and various sugars, sugar alcohols, glucosides, proteins, fats and salts. Four of the amino acids (alanine, valine, leucine and proline) were present in significant quantities. Membrane technology may provide an inexpensive and efficient means for isolation of minor components from stillage.

Some ethanol plants reuse the thin stillage in order to reduce fresh water consumption (Cheryan and Parekh, 1995). The result is that compounds in the thin stillage concentrate over succeeding cycles. Cheryan and Parekh (1995) suggested that if these components could be removed in relatively pure form they would have potential added value. They treated the thin stillage with microfiltration followed by electrodialysis and crystallization. They were able to separate a pure glycerol product as

well as other organic acids. However, they stated that commercial development would depend on creation of markets and ability to compete with compounds currently in use.

Besides ethanol and carbon dioxide, glycerol is one of the major byproducts of corn fermentation (Busche et al., 1992; Julian et al., 1990; Oura, 1977). Glycerol has over 1000 uses in pharmaceuticals, cosmetics, foods, explosives, textiles and other industries, and the world market is greater than a billion pounds a year. Although glycerol from plant sources has not been competitive with petrochemically produced glycerol up until present, this may change with the continued depletion of fossil fuels (Vijaikishore and Karanth, 1986).

It has been proposed that the profitability of an ethanol plant struggling to survive, could be improved by slightly reducing ethanol production in order to generate a glycerol stream. Ethanol production necessarily diminishes because of the consumption of sugar in the glycerol producing process. In addition, further capital investment would be required.

Methodology to recover glycerol from stillage is under development (Keim and Venkatasubramanian, 1989). Julian and coworkers (1990) found that when corn thin stillage was recycled through 5 consecutive fermentations, ethanol yield did not increase but glycerol concentration grew from 0.8% in the original thin stillage to a maximum of 2.1%. Jian and Liu (1991) suggested that glycerin could be recovered from the fermentation media by filtering, vacuum evaporation, vacuum distillation with inorganic powder, decolouration and deodorization. They reported a recovery rate of industrial grade glycerin of 95%.

3.4.7.1.3 Use as a growth medium

Stillage or corn steep liquor has been proposed for use as a medium to produce a number of other products including riboflavin, enzymes such as amylase, invertase or glucose oxidase or antibiotics such as penicillin (Chan et al., 1991, 1992; Maiorella et al., 1983; Linko and Linko, 1981). Steepwater, which contains the soluble compounds, when dried is composed of approximately 50% crude protein (Reiners et al., 1973). It could be further processed to yield methane or ammonia. Amartey and Jeffries (1994) used corn steep liquor as a nutrient source when fermenting D-xylose (found in hemicellulose from corn fibre and hulls) to ethanol. They found that corn steep liquor provided a good and inexpensive source of nitrogen, vitamins and other nutritional factors essential to the activity of the fermenting yeast.

Paik and Glatz (1994) suggested that although propionic acid is generally produced from petroleum feedstocks, production from corn steep liquor merits consideration. Propionic acid is used in thermoplastics, antiarthritic medicines, perfumes, flavours, solvents and as an antifungal agent in foods and feeds. Corn steep liquor, as a byproduct of fuel ethanol production, represents a potentially abundant and inexpensive substrate for fermentation by propionibacteria. Paik and Glatz (1994) achieved concentrations of 45.6 g/litre of propionic acid using fed batch fermentation and immobilized cells. Production of acetic acid using corn steep liquor was also proposed.

3.4.7.2 *Carotenoids*

Hayman and coworkers (1995) indicated that selected coproducts from fuel ethanol plants have potential as microbial growth media for the production of value-added products. They tested six outputs of the commercial wet milling process as substrates for the growth and carotenoid production of *Phaffia rhodozyma*. Carotenoids are valuable pigments used in poultry and

aquaculture feeds to achieve colours necessary for consumer acceptance. Biologically produced carotenoids, such as astaxanthin which is synthesized by *P. rhodozyma*, could provide an alternative to synthetic compounds currently in use, if they could be produced economically.

Hayman and associates (1995) found that use of thin stillage, condensed distillers' solubles and corn gluten feed resulted in the highest accumulation of biomass and carotenoids. Since thin stillage and condensed distillers' solubles have the least potential for economic recovery, they would be the media of choice.

3.4.7.3 *Pullulan*

Leathers and Gupta (1994) looked at the potential use of corn wet milling byproducts as media for the production of pullulan by *Aureobasidium sp.*. Pullulan is an industrial biopolymer that is used in food, pharmaceuticals and other industries and is useful as a film for coating and packaging food and as a low calorie ingredient (Yuen, 1974). At present, pullulan is produced from petroleum products. While *Aureobasidium sp.* was found to grow well on both corn fibre and condensed distillers' solubles, pullulan was not produced when corn fibre was used (Leathers and Gupta, 1994). Use of condensed distillers' solubles, that sell for as little as $0.01 U.S. per pound, did result in pullulan production. The biopolymer was separated from the culture supernatant using organic solvents. In theory, ethanol produced on-site at a biorefinery could be utilized as the organic solvent. The diluted ethanol from the pullulan precipitation could be recovered by distillation. The key to commercialization will be economic competitiveness with current sources of pullulan.

4. OATS

4.1 Introduction

While wheat, corn and barley have received the most attention as feedstocks for fuel ethanol production, there is a number of reasons why oats could be considered as a substrate, with minor adaptations of technique. Oats have a higher yield of starch on a per hectare basis compared to wheat, and can be grown in poorer conditions and soil (Thomas and Ingledew, 1995). Oat yield on a per hectare basis is also higher than for barley or corn. Dale (1991) reported that ethanol yield from oats ranges from 580-1,160 litres/ha/year. This is greater than for wheat (510-710 litres/ha/year) or barley (300-625 litres/ha/year), but less than for corn (600-1,940 litres/ha/year). Therefore, even though ethanol yield from oats, on a grain weight basis, is lower than for wheat, barley or corn (240 litres/ton compared to 340, 250 and 360 litres/ton, respectively), ethanol yield per hectare compares favourably (Dale, 1991).

4.2 Oat Composition

Oats come in many forms and can be grown under a wide variety of conditions. Hulled or covered oats, those with the lemma and palea attached, are the most common. Protein content of whole oats ranges from 15-22% (Cluskey et al., 1979). Oats have a good nutritional value, especially in terms of lysine content. Compared to other cereals, oat groats (hull removed) tend to have higher percentages of protein and fat than other cereals, while carbohydrate content is lower.

The percentage of hull in oats varies from 20-40% (Caldwell and Pomeranz, 1973). While the hull is generally included in animal feeds, it is removed for human and industrial uses. Hulless oats, more recently developed, tend to have lower yields and are not as widely grown. Thomas and Ingledew

(1995) compared the chemical composition of hulled and hulless oats. For the two lines they studied, the hulless strain had higher contents of starch (60%), protein (16%), lipids (6%), and ß-glucan (6%) and lower ash (2%) than the hulled variety (51%, 11%, 5%, 3% and 3%, for starch, protein, lipids, ß-glucan and ash, respectively). Paton and associates (1995) listed the composition of oat groats as 13-20% protein, 55-64% starch, 6-9% lipid, 7.5-12.5% total digestible fibre (TDF) and 3.5-5% ß-glucan. They also reported on the protein, starch, lipid, TDF, ß-glucan and ash composition of a number of oat byproducts including low-bran oat flour, high-purity oat starch, spray-dried oat protein, coarse bran and ß-glucan.

Caldwell and Pomeranz (1973), indicated that the oat groat itself, is made up of the pericarp and testa which form 3% of the kernels' weight, the aleurone which is a single layer of cells comprising 6-8% of the kernels weight, the starchy endosperm accounting for 50-55% and the embryo with approximately 3% of the weight.

4.3 Production of Ethanol from Oats

Oats have not traditionally been used as a feedstock for ethanol production (McCurdy, 1986). However, in recent years a number of value-added products have been extracted from oat grains that have application as functional foods or nutraceuticals. There is potential for ethanol to be produced as a coproduct of the extraction of these more valuable components.

Thomas and Ingledew (1995) studied the processing of hulled and hulless oats into very high gravity (VHG) mashes that could be readily fermented to form ethanol. One of the problems associated with the use of oats in ethanol fermentation is the formation of gels that occur as a result of the solubilization of ß-glucan and pentosans, both found in high concentrations in oats. Gel formation was alleviated by the use of enzymes and by reducing the water to grain ratio. Thomas and Ingledew concluded that oat mash has excellent potential as a feedstock for fuel ethanol production.

Burrows and coworkers (1984) patented a process for the fractionation of oats into the endosperm and bran fractions. This process was further refined by Collins and Paton (1991, 1992). Their procedure involved the treatment of cereal grains, particularly oats, in order to recover the bran fraction, the flour and other components. The latter, though found in low concentrations, had high potential monetary value. In Collins' and Paton's process, the grains were steeped in water and SO_2 in order to liquify the endosperm, then macerated with ethanol. Screens with different mesh sizes were used to produce bran and flour fractions. Their technique could be applied to wheat or rye as well, but not to barley or corn. One of the attributes of the process was the extraction of the ß-glucan component with the bran. This combination material had similar properties of viscosity and flow behaviour to isolated oat gum, but was more economical to produce.

Collins and Paton were able to recover a number of products with value-added potential from the alcohol extract, using anionic exchange columns. These products included phenolic acids, alkaloids, fatty acids, organic acids and amino acids.

4.4 Potential Coproducts of Ethanol Production from Oats

4.4.1 Introduction

Paton and coauthors (1995) used a flow-chart to illustrate the fractionation of oats into its component parts. The groats can be separated into three streams: coarse bran, fine bran and low bran

flour. From the coarse bran, ß-glucan can be extracted, while the low bran flour can be further processed to yield starch and protein.

4.4.2 Protein

While oats have not been wet milled on an industrial basis, wet extraction methodology has been developed in order to produce protein concentrates. Cluskey and associates (1979) reported that when oats were subjected to a wet milling process, a protein concentrate with a good amino acid profile, a bland flavour, plus reasonable hydration capacity and emulsion stability was produced. The product was suggested for use in preparation of protein fortified milk-like and breakfast-type beverages, prepared foods, baked goods, breakfast cereals, crispy extruded snack products and as a meat extender (Cluskey et al., 1976, 1979). A water-soluble gum fraction, with a protein content of approximately 15%, was also produced. Suggested potential uses of the gum included a replacement for eggs in cookies, a thickener, a stabilizer for ice cream, fabric sizing or in pharmaceuticals. The authors felt that the potential for use of oat protein concentrates and gums was very high, but limited by production capacity.

Wu and coworkers have written a number of papers describing the potential use of oats in manufacturing protein concentrates (Wu, 1990; Wu and Stringfellow, 1973; Wu et al., 1972, 1973, 1977). Satterlee (1981) reported that, when measured by the PER assay, oats had greater nutritional quality than the other small grains, even though they are limiting in the three essential amino acids - lysine, threonine and methionine.

Wu et al. (1973) produced an oat protein concentrate using a wet milling process to separate the groat into starch and protein. The cultivar of oat was found to play a role in the final nutritional value of the concentrate, some varieties being of higher protein concentration than others.

Ma (1983a,b) also prepared oat protein concentrates. Using a wet milling technique and two cultivars, one with a high-protein content and another with a medium-protein content, he produced concentrates with 60-70% protein. Functional properties of the concentrates, including foaming ability, fat-binding capacity, solubility and emulsifying properties compared favourably with wheat gluten and soy protein isolate. Ma concluded that if a market niche for the particular functional properties of oat concentrates were found, they could be competitively produced when compared to other phyto-protein concentrates.

Two problems associated with the production of high protein concentrates from residual fermentation broth are the high cost of energy used to evaporate the stillage and the degradation protein by heat. Wu (1990) took ground oats, groats and oat flour and fermented them to form ethanol. The stillage that resulted after the ethanol was removed was divided into distilled grains, centrifuged solids and stillage solids. For each kilogram of ethanol that resulted, 7 litres of oats stillage or 5.4 litres of oat flour stillage was produced. Wu (1990) used reverse osmosis and ultrafiltration to concentrate the material, with significantly less energy input. Depending on the initial substrate, the products included oat distillers' grains with a moderate protein and a high dietary fibre level, or oat flour distillers' grains and centrifuged solids that contained lower levels of dietary fibre, but more protein. Wu (1990) indicated that both products should have value-added potential in the food processing industry.

Oats are used by the primary alcohol producer in Finland, Alko, Ltd. to produce a high-protein oat flour in conjunction with ethanol production (Lapveteläinen and Aro, 1994). Dietary fibre, useful in

cereal products, is removed from the groat and the resultant slurry is separated into starch and protein. The high-protein oat flour is produced by dewatering the protein fraction, washing the protein to remove undesirable flavours and then spray drying it. The final product has a protein content of about 55%, three times that of intact oat groats. Lapveteläinen and Aro (1994) found that the processing protocol did not significantly alter the amino acid composition and molecular weight distribution of the protein in the flour, compared to the original groats. When compared to commercial soy protein concentrate, the oat product had similar solubility and emulsifying properties, but absorbed less water over the pH range considered.

Oat protein also has non-food uses, such as in the cosmetics industry. Paton and co-authors (1995) discussed the use of hydrolysed oat protein in shampoos. Hydrolysed oat protein has a good amino acid content for hair conditioning and has less odour than other protein hydrolysates.

4.4.3 Distillers' Dried Grains (DDG), Distillers' Dried Solubles (DDS) and Distillers' Dried Grains with Solubles (DDGS)

DDG are produced when grain is fermented to form fuel ethanol using the dry milling process. Protein, fat and fibre are enriched in the byproduct, because of the removal of the starch. Kim and associates (1989) considered the potential use of DDG from a number of cereals, including oats, to enhance the nutritional value of extruded snack foods. From their studies, they concluded that oats had the least potential for use, based on torque, density, yield and longitudinal expansion. Oat DDG had the highest fibre and lowest protein content of the DDG evaluated, which probably accounted for its poor performance.

4.4.4 Oat Starch

Canamino Inc., a Saskatoon-based company, is marketing oat products for the cosmetic industry and for human food (Paton et al., 1995; Bioenergy West, February 1994; Forward, 1994). They have developed an oat starch product that takes advantage of recent concerns about the safety of talc. The oat starch product can be used in dusting and baby powders, antiperspirants, blush and eye shadow. Other Canamino products include ingredients for lotions, creams, gels, shampoos and sunscreens as well as modified amino acid compounds that facilitate application, adherence, and water repulsion. Liposomes and phospholipids, found in the groat, may aid movement of vitamins directly into the skin and act as moisturizers. The lower quality starch component that does not have a value-added use, can be fermented into ethanol and used to fractionate the original grain or for fuel.

4.4.5 Oat Hulls

Oat hulls may have potential use as a byproduct of the ethanol production process if the groats are dehulled before fermentation. The hulls contain significant amounts of cellulose, that requires specialized fermentation technology still under development (McCurdy, 1986). Industrial uses of oat hulls are discussed by Caldwell and Pomeranz (1973). These include the manufacture of furfural, adhesive chemicals, abrasives, filters and cavity preventors for the dental industry.

4.4.6 Minor Components

4.4.6.1 *Non-starch Polysaccharides*

A review of the early work done on the non-starchy polysaccharide contents of oats was done by

D'Appolonia (1973). Salomonsson and coworkers (1984) reported on an initial study of the chemical characteristics of the small grains, including oats, focussing on potential for ethanol production or other industrial non-food uses. Particular attention was paid to the non-starch polysaccharide contents of the grains. This was because they could see the potential for operating a fuel ethanol plant with a value-added fibre coproduct stream on the side. Oats were found to have the highest dietary fibre content of the small grains.

Clark (1972) discussed a wet milling process to separate the protein-starch-gum fractions of the oat groat. This was done on a pilot-plant basis. Oat gum originates from the endosperm and is a hot water-soluble polysaccharide that can cause problems in the fractionation of protein and starch in the wet milling process. Oat gum, which is composed mainly of (13)(14)-ß-D-glucan (Wood et al., 1989a), accounts at least partially, for the viscosity or gelatinous nature of cooked oatmeal cereal, and for the emollient effects when used for cosmetics. Studies at Agriculture and Agri-Food Canada originally intended to investigate the production of concentrated protein products for animal consumption, are now focussing on this so-called byproduct of the process, because of its unique qualities and because of lack of anticipated demand for the feed product (Wood et al., 1989a).

ß-glucan is also found in significant quantities in the bran fraction of the oat groat. Wood and associates (1989a) described a procedure used for fractionating oat bran and gum from the intact grain. It is interesting to note that ethanol was used in some of the extraction procedures. Again, this lends itself to the idea of a biorefinery with different product streams where ethanol might be processed for the fuel industry, but also used to produce value-added chemicals in a different part of the plant.

Wikström and coworkers (1994) produced highly pure (88-99.5%) extracts of ß-glucan from the bran and the endosperm of the oat groat in order to compare their rheological properties. They found that viscosity of the isolate from the bran was five times greater than that from the endosperm and that viscosity was not affected by heat treatment.

Oat products have been used for skin care for many years, long before any of the active components were scientifically characterized (Paton et al., 1995; Caldwell and Pomeranz, 1973). When oat bran is finely pin-milled, it exhibits colloidal properties that make the bran useful as a skin protectant and moisturizer (Paton et al., 1995). Oat ß-glucan is effective as a stabilizer and a thickener in moisturizing formulations, and has been used in after shave lotions and face creams.

Potential pharmacological properties of oat products have been discussed for several decades. ß-glucan, the most well known component, has been linked to treatment of heart disease and diabetes. deGroot and associates (1963) were one of the first groups to link the consumption of rolled oats to reduced cholesterol levels. However, it was not until 1988, when a well publicized paper by Kinosian and Eisenberg was published, that an unanticipated demand for oat bran resulted (Wrick, 1993). Kinosian and Eisenberg's paper associated the consumption of oat bran with reduced risk of heart disease because of a lowering of serum cholesterol. Demand has lessened in recent years as research has indicated that the effects are only found for subjects with above normal cholesterol levels (Ripson et al., 1992). Wrick (1993) discusses the importance of proper marketing when dealing with a nutraceutical product such as oat bran. She indicates, that if oat bran were marketed such that even a small percentage of those with high cholesterol were consuming the recommended daily amount, product sales would be much higher than they are today.

The link between oatmeal and diabetes was made even earlier (Allen, 1913 in Wood et al., 1989a).

Wood and associates (1989a) indicated that the mode of action of oat bran in reducing postprandial glycaemia has still not been completely ascertained, though viscosity seems to be involved. They have found differences in the molecular weight and viscosity of the ß-glucan depending on processing methodology and indicate that possible activity of other components in the bran may affect results (Wood et al., 1989b).

4.4.6.2 *Phenolic acids*

Collins (1989) indicated that characterization of the phenolic compounds found in oats would be of use in developing oat-based ingredients for use in animal feed and human food. Cereal grain phenolics are involved in a number of biochemical, medical and nutritional processes. Collins and his associates, have identified a number of phenolic acids in oat groats and hulls in recent years, including avenanthramides and hydroxycinnamic acids (Collins, 1986, 1989; Collins and Mullin, 1988; Collins et al., 1991). Cinnamates in wheat have been noted for their redox action and potential for use in sunscreens.

5. BARLEY

5.1 Introduction

Barley has been used as a feedstock for fuel ethanol production in Canada, the United States and Sweden (McCurdy, 1986). Morris (1983) reported that three plants were using barley to produce ethanol in the U.S., but that their total production was small, representing less than one percent of the national output. Mohawk Oil's plant in Minnedosa, Manitoba used contract or utility barley for at least one year before it switched to a mix of 80% corn and 20% barley because of viscosity problems in the mash (McCurdy, 1986). St. Lawrence Reactors reported an ethanol yield of 367 litres/tonne with a byproduct output of 312 kg/tonne based on a 90% dry matter when barley was the feedstock considered, while Dale (1991) listed a yield of 250 litres per ton or 300-625 litres per hectare per annum.

5.2 Composition of the Barley Grain

Pomeranz (1973) reported that barley contains on average 63-65% starch, 8-13% protein, 2-3% fat, 1-1.5% soluble gums, 8-10% hemicellulose and 2-2.5% ash. Hulless barley lines, high in both protein (particularly lysine) and starch, and low in fibre, have recently been developed. Ingledew et al. (1995) found that milling, mashing and fermenting of hulless lines were less difficult than for hulled cultivars and that addition of beta-glucanase alleviated viscosity problems. The distillers' grains that remained after the distillation of the ethanol had protein concentrations comparable to wheat distillers' grains. Protein contents were higher than for hulled barley varieties and non-digestible fibre concentrations were lower.

Since starch recovery from barley is not as high as that from corn, if ethanol production is going to be profitable, byproduct recovery becomes even more essential (Wu, 1985, 1986). The nutritional value of barley, based on amino acid content, is greater than that for corn and is not significantly affected by the fermentation process.

5.3 Ethanol Production from Barley

McCurdy (1986) reported that very little information exists on the wet processing of barley to

separate protein and starch. It should be possible to adapt the methodology developed for wheat, however. Barley would not have the processing problems associated with wheat gluten (Munck, 1981), but may have its own unique challenges associated with its beta-glucan content (McCurdy, 1986). Thomas and coworkers (1995) reported on the production of fuel ethanol from hulless barley using VHG fermentation technology.

5.4 Potential Coproducts of Ethanol Production from Barley

5.4.1 Protein

Wu and coworkers (1979) looked at the production of protein concentrates from high-protein, high-lysine barley cultivars. Using alkaline solubilization and acid precipitatin techniques, they were able to isolate 57-77% of the original protein. Hydration capacity of the protein was satisfactory, but emulsion properties were poor.

Wu (1985, 1986) looked at the potential for extracting a protein concentrate from barley fermentation stillage using ultrafiltration and reverse-osmosis. Wu concluded that there was potential for distillers' dried grains and centrifuged solids to be used for human consumption. Protein contents for barley distillers' grains and centrifuged solids were 32.6-36 and 60.5-67% (dry basis), respectively. This compares to approximately 13% for the original grain.

5.4.2 Fibre

Barley, like oats, may contain dietary fibres that would be of benefit to health when consumed by humans (Wood et al., 1989a; Salomonsson et al., 1984). While most barley cultivars contain amounts of ß-glucan comparable to that found in oats, some varieties have been found to have enriched quantities of ß-glucan, at levels similar to that found in just the oat bran fraction (Åman and Graham, 1987).

Insoluble dietary fibre-rich components (lignin and cellulose) from barley have been linked to cancer prevention in rats (McIntosh et al., 1993). In their study, McIntosh and associates found that when intestinal cancer was chemically induced in the rats, dietary spent barley grain was more protective against the disease than wheat bran or barley bran rich in soluble fibre.

San Buenaventura and associates (1987) looked at the total dietary fibre (TDF) content of barley dried distillers' grains with solubles (DDGS) to ascertain the potential for use in human food as a dietary fibre additive. They found that TDF was concentrated almost four times in the barley DDGS compared to barley itself, with approximately 85% of barley DDGS weight attributable to fibre. This was more than twice the fibre found in whole wheat and also significantly greater than that found for DDGS from wheat, corn or brown sorghum. They concluded that DDGS represent a potential source of dietary fibre for use in human food.

5.4.3 Distillers' Dried Grains (DDG), Distillers' Dried Solubles (DDS) and Distillers' Dried Grains with Solubles (DDGS)

Kim and associates (1989) considered the potential for inclusion of distillers' dried grains from barley in extruded grain products. DDG is high in fibre, protein and fat and therefore could add nutritional properties to extruded snack foods that are not generally renowned for their nutritional value. Kim and coworkers found that barley DDG was not as effective as corn or wheat, but was more effective

than oats, when evaluated on the basis of torque, density, yield and longitudinal expansion.

One of the problems associated with the utilization of barley distillers' grains in food is the flavour (Dawson et al., 1987). This is due at least in part to the level of fat, which similar to protein, is concentrated in the stillage fraction (Wu, 1986). Dawson and coworkers (1987) tracked the changes that occurred in the neutral lipids as barley grain underwent the production process to produce fuel ethanol. They found that major alterations in the lipids occurred, namely saturated fatty acids increased and unsaturated fatty acids decreased. Taste panel evaluations of granola and granola bars containing no barley, ground barley, full fat barley dried distillers' grains or defatted barley dried distillers' grains, indicated that the inclusion of barley dried distillers' grains in granola had negative effects on flavour acceptability regardless of defatting. In previous experiments, Dawson and associates (1984) reported that defatted DDG, from barley, could be incorporated into oatmeal cookies at a 15% concentration without affecting taste panel choice.

5.4.4 Minor Components

5.4.4.1 *Enzymes*

-amylase, -amylase inhibitors, ß-amylase and oxalate oxidase are minor components found in barley that may have potential for extraction and commercial use (Forward, 1994; Murray et al., 1987). Since -amylase is currently used in the malting industry, a market already appears to exist. -amylase inhibitors, which are water-soluble proteins, are commercially available and may have future potential for use in sprouting prevention in wheat. The market for oxalate oxidase is not very large at the present time. However, there is some scope for expansion, which may justify isolation from barley in the future. Unfortunately, Forward (1994) predicted that none of these components had a high potential for extraction in the very near future.

5.4.4.2 *Tocols*

Tocols (tocopherols and tocotrienols) have been identified as another minor component in barley with value-added potential. Peterson and Qureshi (1993) reported that barley is one of the prime sources of tocols. More than 2000 papers have been published on the clinical attributes of tocophenols (Weber, 1973). Not all claims can be substantiated but some, such as the ability to lower serum cholesterol (Qureshi et al., 1991; Weber et al., 1991) and to act as an antioxidant (Burton and Traber, 1990), have credibility.

Peterson (1994) found that because milling or brewing of barley concentrated tocol levels, the byproducts had potential for use in food products used by people attempting to lower their serum cholestrol through diet. Weber and coworkers (1991) recently reported that ingredients from brewers' spent grain had a positive effect on lowering cholestrol levels.

5.4.4.3 *Citric Acid*

Myung and coworkers (1992) reported on the use of alcohol distillery wastewater (barley as a feedstock) as a substrate for citric acid production. Two purposes are served through this use; solution to a waste disposal problem and production of a value-added material. *Aspergillus niger* ATCC 9142 was used to treat the wastewater from the fermentation of hulless barley to produce alcohol. When the wastewater had a 50 g/litre reducing sugar concentration, treatment resulted in a 2.4 g/litre citric acid concentration in the batch reactor. The COD (mg/litre) (i.e. polluting potential)

of the final effluent was reduced by approximately 39%.

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Chapter 2. Current Utilization of Coproducts and Near Coproducts of Ethanol Fermentation from Grain

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1. CURRENT STATUS OF COPRODUCT UTILIZATION IN NORTH AMERICA

1.1 Introduction

At present, the coproducts produced by most ethanol plants in North America are used in the animal feed industry. These include distillers' dried grains (DDG), distillers' dried grains with solubles (DDGS), distillers' dried solubles (DDS), wet distillers' grains and other types of animal feed ingredients. Some of the larger plants in the U.S. are producing a wider variety of materials including wheat gluten, corn gluten, corn germ, corn oil, carbon dioxide (CO2) and food-grade yeast. Many, though not all, fuel ethanol firms are interested in pursuing value-added potential for their byproducts. Some are actively engaged in research while others are considering what steps to take in order to become involved in this area.

Wherever possible current research interests of particular ethanol firms are noted in this present report. A number of companies were willing to give information concerning feedstocks and coproducts, but would not discuss research efforts. The information presented here came from a number of sources including direct contact with producers and in published material: Powers (1995), Mulligan (1994), ICAST (1994), Monenco AGRA (1993), the New Uses Council (USA) and the American Renewable Fuels Association.

1.2 Canadian Ethanol Plants

1.2.1 Plants in Operation

Commercial Alcohols Inc. uses corn to produce ethanol at its Tiverton, Ontario plant. Distillers' wet grains, produced as a byproduct of the process, are sold for animal feed. Commercial Alcohols has planned an expansion project at Chatham, Ontario. Groundbreaking was projected for the spring of 1996, but may be delayed until mid-1997 because of financial considerations. At their current site, Commercial Alcohols has considered extracting CO2 for sale to a local greenhouse operation, but this project has not come to fruition. CO2 extraction is also being considered at the Chatham plant.

Mohawk Oil Co. Ltd. currently sells its wheat-to-ethanol fermentation byproducts for animal feed. However, they will soon be producing a food-grade coproduct called Fibrotein at their Minnedosa, Manitoba plant. It has taken three years to obtain the Canadian rights to the product, to do market research and to gain regulatory approval. An official announcement is due imminently (Don O'Connor, personal communication).

Pound-Maker Agventures Ltd. of Lanigan, Saskatchewan uses all byproducts on-site for cattle feed. They have just finished an expansion project that allows handling of 24,000 head in their feedlot (Saskatoon Star-Phoenix, 1994). Pound-Maker has also considered use of its waste heat in

greenhouse operations. They are extremely interested in finding higher value uses for their byproducts in order to increase profitability (John McEachran, personal communication).

1.2.2 Plants under Development

Agri-Partners International is developing a wheat biorefinery in Red Deer, Alberta in conjunction with TDI Projects Inc. (Earl St. Denis, personal communication). This plant will produce ethanol, as well as a number of other wheat-based products.

Metalore Resources Ltd., presently involved in the natural gas industry, has planned a $40 million ethanol/food processing plant in Walsh, Ontario (George Chilian, personal communication). Their major feedstock will be hard red winter wheat that they will preprocess for fibre, gluten and germ removal before fermentation to ethanol. Other planned coproducts include fusil oil, CO_2 and distillers' grains. Value-added opportunities for the distillers' grains are being investigated. Metalore hopes to begin construction in the summer of 1996 with production beginning early to mid-1997.

Seaway Valley Farmers' Energy Co-operative was formed by 15 farmers in 1992 (Bud Atkins, personal communication). They are planning a 50 million litre plant in Cornwall, Ontario with start of construction slated for the spring of 1996. Corn will be used as the major feedstock, but hulless oats, wheat and barley are also being considered. They will produce half of their ethanol for fuel and the other half for industrial and food usage. Initial coproducts will include DDGS and CO_2, but accommodation is being made in construction of the plant to allow for other value-added product streams. Seaway Valley's plant will be environmentally friendly, utilizing all components of the feedstock and generating no waste.

1.3 AMERICAN ETHANOL PLANTS

According to the American Renewable Fuels Association, there are 44 ethanol producers in the United States with plant capacities ranging from 750 million gallons per year (MGY) to 0.5 MGY. As of January 1996, the top four producers were Archer Daniels Midland Corn Processing (750 MGY), Minnesota Corn Processors (120 MGY), Cargill (105 MGY) and Pekin Energy Company (100 MGY). A number of the American ethanol plants are listed below. They are arranged alphabetically under type of feedstock and/or process.

1.3.1 Corn - Dry Milled

Company	Location	Coproducts	Coproduct Research
Archer Daniels Midland Corn Processing	Peoria, Illinois	DDG	xanthan gum, lactic acid, amino acids, glycine, membrane technology
Alchem	Grafton, North Dakota	DDG	
AG Processing, Inc.	Hastings, Nebraska	DDG	looking at value-added applications for byproducts
Broin Enterprises Inc.	Scotland, South Dakota	wet and dry animal feed	

Chief Ethanol Fuels, Inc.	Hastings, Nebraska	DDG	
Corn Plus	Winnebago, Minnesota	DDGS	use of processing techniques that separate the corn kernel into fibre, germ, gluten and starch without the steeping process
ESE Alcohol	Leoti, Kansas	fertilizer	
Farmland Industries	Kansas City, Missouri	DDS and solubles	
Heartland Corn Products	Winthrop, Minnesota	DDG	
Heartland Grain Fuels LP	Aberdeen, South Dakota	DDGS, thin stillage, condensed distillers' solubles	
High Plains Corporation	Colwich, Kansas; York, Nebraska	DDG, wet distillers' grains, CO_2	
Manildra	Hamburg, Iowa	DDG, condensed distillers' solubles	
Minnesota Clean Fuels	Dundas, Minnesota	mash from stripper column	
Morris Ag Energy	Morris, Minnesota	DDGS, concentrated solubles	would be interested in investigating the use of membrane technology to extract value-added materials from stillage
Nebraska Energy, L.L.C.	Aurora, Nebraska	DDGS, wet distillers' grains, concentrated solubles	
New Energy Company of Indiana	South Bend, Indiana	DDGS, CO_2	fermentation of corn fibre, membrane extraction of thin stillage and steep water to recover glycerol, lactic acid and other minor components
North Carolina Ethanol	Faison, North Carolina	DDGS, CO_2	
Reyncor	Shreveport, Louisiana	wet distillers' grains	
South Point Ethanol	South Point, Ohio	DDGS, CO_2	glycerol, lactic acid, yeast, corn oil, corn germ, lignin

Vienna Correctional Research Center	Vienna, Illinois	cattle feed

1.3.2 Corn - Wet Milled

Company	Location	Coproducts	Coproduct Research
Archer Daniels Midland Corn Processing	Decatur, Illinois; Cedar Rapids, Iowa; Clinton, Iowa	gluten feed and gluten meal	xanthan gum, lactic acid, amino acids, glycine, membrane technology
A.E. Staley MFG. Co.	Loudon, Tennessee	high fructose corn syrups	
Cargill	Eddyville, Iowa; Blair, Nebraska	corn oil, corn gluten feed, high fructose corn syrup, (regular corn syrup, citric acid and sodium citrate - Eddyville only)	
The Hubinger Company	Keokuk, Iowa	stillage for animal feed	
Minnesota Corn Processors	Marshall, Minnesota; Columbus, Nebraska	main process is the generation of corn starches and sweeteners	
Pekin Energy Company	Pekin, Illinois	corn gluten feed, high protein gluten meal, corn germ	product improvement, glycerol recovery, membrane technology

1.3.3 Wheat

Company	Location	Coproducts	Coproduct Research
Alcotech	Ringling, Montana	DDG, soluble protein recovery using membrane technology	
American Ethanol Corp.	Coeur d'Alene, Idaho	vital wheat gluten, CO2, DDG	
Manildra	Hamburg, Iowa	vital wheat gluten	
Minnesota Clean Fuels	Dundas, Minnesota	animal feed	

1.3.4 Barley

Company	Location	Coproducts	Coproduct Research
American Ethanol Corp.	Coeur d'Alene, Idaho	CO_2, DDG	

2. POTENTIAL VALUE-ADDED OPPORTUNITIES FOR ETHANOL COPRODUCTS

2.1 Introduction

A number of cereal derivatives have been identified that are currently used in cosmetics, food and other industries. In some cases, the specific chemicals present in wheat, oats, barley or corn could replace synthetic compounds already in use in the market place. The limiting factors include low concentrations, development of economical extraction techniques, cost-competitiveness with compounds already being used, market volumes and production-to-market mechanisms (Christensen, 1994). So far, interest has weighed more heavily on the government/producer end of the equation than on the market place end.

Tables listing concentrations of the chemical components of wheat, oats, corn and barley can be found in the Food Composition and Nutrition Tables (1989/1990). In addition to the major components such as starch, protein and fat, minor components including amino acids, tocopherols, tocotrienols and vitamins are recorded.

Le Jardin de l'Aigle Reg. (1994) surveyed the potential coproducts from nineteen species of crop plants, including barley, oats and durum wheat, for Agriculture and Agri-Food Canada. The majority of their information came from two American databanks: Natural Products Alert (NapralertTM) generated by the College of Pharmacy, University of Illinois, and the Agricultural Research Service, USDA, Beltsville, Maryland. In their report they listed all of the known compounds found in these crops based on the scientific literature. Where possible, they linked these chemicals to their present use in cosmetics, pharmaceuticals, foods and other industries. In summary, Le Jardin de l'Aigle identified fourteen minor components found in oats and/or barley and evaluated them for coproduct potential. Some components are also found in wheat and corn. The list included amino acids (alanine, arginine, glycine, glutamine and tyrosine), vitamins (vitamin B1, vitamin B2, pantothenic acid, niacin, vitamin E (-tocopherol) and folic acid), as well as a number of other materials including ß-carotene, diethylamine and piperidine. These compounds will be discussed in more detail in the following section. However, Le Jardin de l'Aigle concluded that the potential for commercial development of these compounds as coproducts from fuel ethanol production was small.

Murray and co-workers (1987) reviewed the minor components that can be found in a number of agricultural resources including wheat, oats, corn and barley. Since the completion of their study, Dr. Murray has had over 1000 requests for copies of the report (Don Murray, personal communication). While there has been a significant amount of interest in minor component recovery, Dr. Murray indicated that he was not aware of many instances where research had been followed through to commercialization.

An extensive review of the cereal processing industry was done by Tam McEwen (1995) of Threshold Technologies Company for Agriculture and Agri-Food Canada's Winnipeg Policy Branch. McEwen discussed a number of products that can be derived from the fractionation of several grains including oats, wheat, and barley. The report emphasized the need for collaborative activity between industry and researchers, and identified a number of cereal fractionation opportunities. At present,

there are no wheat preprocessing facilities operating commercially in Canada. While preprocessing technology has not been developed specifically for fuel ethanol production, it could easily be added to an ethanol plant.

The cosmetics and toiletries industry alone comprises a $60 billion market worldwide and uses approximately 4,000 different additives (Flick, 1991). A number of cereal derivatives are already being produced commercially and used in cosmetic formulations by many companies including Active Organics, Eastman, Laboratoires Sérobiologiques, McIntyre, R.I.T.A, Roche, and Universal Preserv-A-Chem, (Ash and Ash, 1994). Opportunities are enhanced by the recent development of the "green" cosmetics market, as consumers search for environmentally friendly bio-products. The cosmetics and toiletries industry uses many types of raw materials including emulsifiers, emollients, preservatives, binders, stabilizers, wetting agents, dispersants, foaming agents, pearlizers, gelling/stiffening agents, surfactants, and viscosity builders (Flick, 1991), categories where cereal-derived compounds can often play a role.

2.2 Cereal Components with Value-Added Use and/or Potential

2.2.1 Protein Products and Derivatives

Protein is a major constituent in the small grain cereals. In addition to its nutritional value, it has application in the cosmetics and toiletries industry.

2.2.1.1 *Wheat*

Wheat gluten consists of a number of proteins, particularly gliadin and glutenin. The gluten is used in cosmetic powders and creams as a base (Winter, 1994). In the food industry, wheat gluten is used as a dough conditioner, formulation and processing aid, nutritional supplement, stabilizer, surface-finishing agent, texturizing agent and thickener (Lewis, 1989). The International Wheat Gluten Organization describes the use of gluten in a number of products including baked goods, breakfast cereals, meat, fish and poultry products, pasta, pizza, snack foods, tortillas, batter mixes and coatings, pet foods, aquaculture feeds, chewing gum, beverages, biodegradable surfactants and pressure-sensitive adhesive tapes.

Wheat germ protein is used in shampoos and emollients (Winter, 1994). Wheat amino acids are sold under the trade name HydrotriticumTM WAA (Ash and Ash, 1994).

Lectins are carbohydrate binding proteins found in wheat germ. They have a broad range of medical and biochemical uses based on their capacity to bind erythrocytes (Murray et al., 1987). Lectins can be bound to other molecules such as biotin or peroxidase to increase their value. One lectin, agglutinin, can be extracted by affinity chromatography and is sold by a number of biochemical companies for as much as $1700 U.S. per gram.

2.2.1.2 *Corn*

Zein, a protein extracted from corn gluten with an alcohol, is used as a fat mimetic, a glaze and a surface finishing agent for foods and pharmaceuticals (Cook and Shulman, 1994; Lewis, 1989; Wilson, 1987). It is also used in face masks and nail polishes as a plasticizer (Winter, 1994).

Zein can be used to make textile fibres, plastics, printing inks, varnishes and other coatings and

adhesives. Aqueous ultrapurified zein (UPZ) latex formulations can be prepared and have a variety of uses in foods and pharmaceuticals in instances where flammable organic solvents are not desirable (Cook and Shulman, 1994).

Corn gluten amino acids are found in the commercial product Amino Gluten MG, while corn gluten proteins are found in Press-AidTMXF and Press-AidTM (Ash and Ash, 1994). Corn-ProTM35 is a hydrolysed corn protein produced by Brooks Industries for use in the cosmetics industry in skin and hair care products.

2.2.1.3 *Oats*

Different oat plant derivatives have been used in cosmetic and toiletry products because of their gluten content and resultant effects on dry and itchy skin. Commercial bath products, including soaps and gels as well as powders, are produced. Oat gluten is also used as a stabilizer, emulsifier and food extender (The Lawrence Review of Natural Products, 1991). Oat protein is marketed by one company under the trade name MicroatTM (Ash and Ash, 1994).

Another company, Canamino Inc., produces Hydrolysed Oat Protein HOPA for use in shampoos, conditioners, lotions, creams and gels. It has advantages over other plant proteins and because of its amino acid balance, can also replace animal proteins. On skin and hair Canamino's product provides a thin protective layer that retains moisture and provides shine.

2.2.1.4 *Amino Acids*

The protein components of different small grain cereals contain characteristic amounts of various amino acids. Some of these components have known usage in cosmetics, foods, pharmaceuticals and other industries. Whether or not it would prove profitable to extract and market amino acids as coproducts of ethanol production is not known.

Alanine -- Alanine has been identified in oats, corn and wheat. L-alanine is utilized in the pharmaceutical industry, in herbicides and as a moisturizer in cosmetics (Ash and Ash, 1994).

Arginine -- Arginine is an essential amino acid found in wheat, oats, corn and barley. It is used in various cosmetics (Ash and Ash, 1994).

Glutamic acid -- Glutamic acid, classified as a non-essential amino acid, has been isolated from barley, corn, wheat and oats. It is used as a food flavouring enhancer and in medical and biochemical research (Ash and Ash, 1994). The market for glutamic acid in the form of monosodium glutamate is large and is presently supplied by hydrolysis of plant proteins including wheat and corn gluten and by fermentation of suitable carbohydrates (Merck Index, 1976).

Glycine -- Glycine has been identified in oats, corn and wheat (Food Composition and Nutrition Tables, 1989/1990). It is a non-essential amino acid with applications in food, cosmetics, pharmaceuticals, plastics and paints. Glycine is normally manufactured from gelatin or silk fibroin (Merck Index, 1976).

Tyrosine -- Tyrosine has been found in barley, wheat, corn and oats (Food Composition and Nutrition Tables, 1989/1990). It is used as a nutritive additive (Ash and Ash, 1994).

2.2.2 Fibre and Fibre Derivatives

Small grain cereals contain significant quantities of soluble and insoluble fibre. Both types of fibre have been linked to specific health promoting effects (see Chapter 1, Sections 1.3.4, 2.4.2, 4.4.6.1 and 5.4.2 for more detail).

Wheat bran, oat bran and corn bran are all familiar products in baked goods and breakfast cereals. In an ethanol plant or biorefinery, the bran fraction represents an important coproduct stream. Further refining can yield even more value-added opportunities in the food, pharmaceutical and cosmetic industries.

2.2.2.1 *Oats*

Williamson Fifer Products Ltd. of Louisville, Kentucky produces a 98% oat fibre product for use in bread, diet breakfast and nutritional beverages, yoghurt, soups, gravies, sauces and diet food supplements (LaBell, 1992). The product has a high water absorption capacity that makes it useful for simultaneously increasing fibre and decreasing calorie content.

Alko Foods Division produces an oat fibre ingredient that has an enriched content of ß-glucan and up to twice the total dietary fibre of oat bran (LaBell, 1992). It can be used in baked goods, snack foods and breakfast cereals to provide a light moist texture.

Oat gum is used as a thickener and stabilizer in food and cosmetic products, as an antioxidant in butter, creams and candy, and as a thickener and stabilizer in pasteurized cheese spreads and cream cheese (Winter, 1994). The major component of oat gum is ß-glucan, a water-soluble fibre, that has been linked to control of heart disease, diabetes and cancer. Of the small grains, ß-glucan is found in highest concentrations in oats and barley.

Canamino Inc. produces an oat bran product with a high beta-glucan content called OstarTM CI-B14. This product is for use in cosmetic products as an anti-irritant and provides soothing and healing properties. It is an absorbent, moisturizer and film former.

Canamino Inc. also produces an oat beta glucan product called OstarTM Glucan 1A which can replace hyaluronic acid in skin and hair care products, shaving creams, liquid make-up and bath toiletries to provide a thin film allowing for moisture retention.

Quaker Oats Co. and Heller Seasonings & Ingredients, Inc. are jointly developing and marketing a specially processed oat bran product using patented technology developed in conjunction with Webb Technical Group (LaBell, 1992). The product is mixed with very lean meat to produce low fat ground beef or pork sausages.

Another fat replacer, Oatrim, was developed by Dr. George Inglett and co-workers at the Northern Regional Research Centre (USDA) at Peoria, Illinois (LaBell, 1992). The patent for this product has been licensed by Quaker Oats Co., Rhone-Poulenc, Inc. and ConAgra Speciality Grain Products Co. It can be used in frozen desserts, dairy products, salad dressings, baked goods and meat products to reduce fat content while providing a fat-like texture and mouthfeel.

2.2.2.2 *Barley*

Studies have shown that barley ß-glucan has nutritional and health properties similar to those found for oat ß-glucan. ß-glucan is extracted from barley kernels by Sigma Chemicals (Le Jardin de l'Aigle Reg., 1994).

2.2.3 Vitamins

A number of vitamins are present in the small grain cereals, including vitamin B1, vitamin B2, pantothenic acid, niacin, vitamin E (-tocopherol) and folic acid. Vitamins are utilized as nutritional supplements as well as cosmetics additives (Ash and Ash, 1994; drug facts and comparisons, 1994).

Vitamins B1 and B2 -- Also known as thiamin and riboflavin respectively, vitamins B1 and B2 have been identified in oats, wheat, corn and barley (Food Composition and Nutrition Tables, 1989/1990). Riboflavin, produced synthetically, is used as a nutrient in human and animal diets and as a colourant in cosmetic formulations such as tanning compounds (Ash and Ash, 1994). Thiamin also has cosmetic applications.

Niacin -- Also known as nicotinic acid, niacin is present in wheat, oat, corn and barley kernels. It is used nutritionally and as a rubifacient for cosmetics (Ash and Ash, 1994).

Folic acid -- Folic acid is found in the seeds of corn, wheat, barley and oats (Food Composition and Nutrition Tables, 1989/1990). It acts as a nutritional factor and stimulates production of red and white blood cells (drug facts and comparisons, 1994; Merck Index, 1976).

Pantothenic acid -- Pantothenic acid is one of the B family of vitamins (B5) and occurs widely in the plant kingdom, including the four small grain cereals reviewed in this report (Food Composition and Nutrition Tables, 1989/1990; Merck Index, 1976). It is prepared synthetically for commercial use as a nutritional factor and is also available at a 25% concentration in a natural protein for cosmetics use (Ash and Ash, 1994).

Tocopherols -- (Vitamin E) Tocopherols are produced by the vacuum distillation of edible vegetable oils (Winter, 1994). They are found in wheat germ, oats, corn and barley (TWG Consulting Inc., 1995; Lawrence Review of Natural Products, 1994; LaBell, 1992). Tocopherols are used as antioxidants, emollients and solvents in baby toiletries, deodorants, hair products, essential oils and rendered animal fats. They can be used to prevent "warmed over" flavours in poultry and cooked meats. Tocopherols are also used nutritionally as a dietary supplement in that they protect the fat in body tissue from abnormal breakdown and they aid in the formation of red blood cells, muscle and other tissues. Some research has shown a link with control of heart disease and evidence of aging. Tocopherols are marketed by several companies under a variety of trade names (Ash and Ash, 1994).

2.2.4 Fats, Oils and Lipids

Glycerides -- Glycerides consist of a large class of compounds that are esters of the alcohol glycerine (Winter, 1994). At present they are generally manufactured synthetically. However, they are present in wheat germ as mono-, di-, and tri-glycerides (Ash and Ash, 1994; Murray et al., 1987) and marketed under the trade names Vita-Cos and Wickenol®535. Glycerides are used in cosmetics such as lipsticks, creams, lotions, and pigmented products as texturizers and emollients.

Glycerin (also called glycerol, glycl alcohol) -- Glycerin is an oily fluid produced by adding alkalies

to fats and fixed oils (Winter, 1994). It is found in the stillage of wheat (Sosulski and Sosulski, 1994) and corn (Cheryan and Parekh, 1995). Glycerin is generally produced as a byproduct of soap and fatty acid manufacture. It reacts with fatty acids to form monoglycerides that act as emulsifiers and stabilizers (Duxbury, 1991).

Glycerin can be used as a solvent, humectant, plasticizer, emollient or sweetener and has over 1000 uses in pharmaceuticals, cosmetics, foods (e.g. baked goods, marshmallows, candy), explosives, textiles, packaging and other industries (Lewis, 1989; Merck Index, 1976). The world market for glycerol is greater than a billion pounds a year.

Glycerin helps to keep creamy products soft by absorbing moisture from the air and makes spreading easier (Winter, 1994). Some of the many cosmetic and toiletry products glycerin is found in include hand creams and lotions, hair spray, liquid facial foundations, skin fresheners, toothpaste, rouge, freckle creams, facial masks, perfumes and mouthwashes.

Glycerin is also used in a range of food products including colourings, flavourings (Winter, 1994), liqueurs and confectionaries (Merck Index, 1976). It acts as a heat transfer medium for frozen foods, a crystallization preventor in frozen eggs and yolks, a humectant in dry fruit and an agent for smoothness and body development in chocolate syrups and distilled liquors (Duxbury, 1991). It also may be included in blacking, printing and copying inks, lubricants, elastic adhesives, lead oxide cements, antifreeze, or used as a preservative for printing on cotton fabric or as a nutrient for production of antibiotics.

Corn oil -- In addition to the food industry, corn oil is used in the pharmaceutical (Secondini, 1990) and the cosmetics industries (Ash and Ash, 1994). It is used in emollient creams and toothpaste (Winter, 1994). Ash and Ash (1994) list a number of corn oil products including corn oil (Trade names - Lipex104, Nikkol Corn Germ Oil, Super Refined Corn Oil, Univegoil CRN), corn oil PEG-6 esters (Trade name - Labrafil® WL 2125 CS), corn oil PEG-8 esters (Trade name - Labrafil® WL 2609 BS) and corn oil unsaponifiables (Trade name - ETIZM).

Wheat products -- Ash and Ash (1994) list a number of wheat derivatives that fall into the fat and oil classification. Wheat germ oil and wheat germ oil unsaponifiables are two of these. Wheat germ oil is produced under seven different trade names and used in a number of different cosmetic products. Wheat germ oil unsaponifiables are reported as being present in one product. Wheat lipid oxide is marketed under the trade name MackerniumTM WLE.

2.2.5 Quaternary Ammonium Compounds

Wheat germamidopropyl betaine, wheat germamidopropyl dimethylamine, wheat germamidopropyl dimethylamine hydrolysed collagen, wheat germamidopropyl dimethylamine hydrolysed wheat protein, wheat germamidopropyl dimethylamine lactate, wheat germamidopropyl dimonium hydroxypropyl hydrolysed wheat protein, wheat germamidopropyl silk hydroxypropyl dimonium chloride and wheat germamidopropyl ethydimonium ethosulfate fall into the category of quaternary ammonium compounds (Ash and Ash, 1994; Winter, 1994). Quaternary ammonium compounds are used as preservatives, surfactants and antiseptics and are generally derived synthetically from ammonium chloride. They are found in deodorants, after-shave lotions, shampoos, antiperspirants, cuticle softeners, hair products, hand creams and mouthwashes.

2.2.6 Pigments

Carotenoids, particularly astaxanthin, are used for pigmentation in poultry and aquaculture diets in order to provide colouration expected by the consumer of eggs and certain types of fish (Hayman et al., 1995). ß-carotene, which acts as a yellow colourant in the food industry and as a vitamin A precursor (Merck Index, 1976) is found in barley seeds. Carotene is found in corn and wheat grains (Food Composition and Nutrition Tables, 1989/1990). USDA scientists have shown that the red yeast *Phaffia rhodozyma* is able to grow and produce carotenoid pigments using various ethanol byproducts as substrates, including corn fibre and corn gluten (Hayman et al., 1995). There is industrial interest in exploring commercialization of this technology.

2.2.7 Enzymes

A number of enzymes, now used industrially can be found in the cereal grains. Lipase, for example, is found in wheat germ and bran as well as in oat hulls. It can be used to hydrolyse fats and oils without damaging other constituents including vitamins or unsaturated fatty acids. Lipase is used in the food industry for flavour enhancement and in detergents for the improvement of cleaning action (Merck Index, 1976).

Carboxypeptidase and phytase are also present in wheat bran (Murray et al., 1987). Carboxypeptidase is utilized in amino acid sequencing of proteins. Phytase is used to hydrolyse phytic acid.

Acid phosphatase, sucrose synthetase and sucrose phosphate synthetase have been found in wheat germ (Murray et al., 1987). Sigma Chemicals extracts acid phosphatase from wheat (Le Jardin de l'Aigle Reg., 1994). They also extract -amylase inhibitor from wheat and - and ß-amylase from barley. -amylase inhibitor's have potential for use in the treatment of wheat kernels to prevent sprouting during harvest.

2.2.8 Phytate and Phytate Derivatives

Phytic acid is found in the cereal grains including wheat and corn. It is used as a complexing agent for heavy metal ions and as a sodium salt (Merck Index, 1976). Phytate derivatives have also been suggested for treatment of a number of diseases including cancer, heart disease and diabetes (TWG Consulting Inc., 1995). Phytin® is a phytic acid calcium magnesium salt which is found in a number of grains (Merck Index, 1976). One of the primary sources is corn steep liquor. Phytin® is used as a nutrient, tonic, calcium supplement and as a feedstock for inositol manufacture. For more information see Chapter 1, section 2.4.5.1.

2.2.9 Organic Acids

A number of studies are investigating the production of organic acids from corn-to-ethanol byproducts (Powers, 1995). Organic acids have a number of functions. Acetic acid, propionic acid, butyric acid and lactic acid can be used as food additives or feed preservatives. Lactic acid can be used for the production of biodegradable plastics or in cosmetics (Ash and Ash, 1994). Acetic acid can be used as a feedstock for production of other chemicals (pharmaceuticals, plastics, dyes, insecticides, photography chemicals), as an antioxidant or as the deicer calcium magnesium acetate.

Propionic acid is used in thermoplastics, antiarthritic medicines, perfumes, flavours, solvents and as an antifungal agent in foods and feeds (Paik and Glatz, 1994). Corn steep liquor, as a byproduct of

fuel ethanol production, represents a potentially abundant and inexpensive substrate for fermentation by propionibacteria.

2.2.10 Extracts

Ash and Ash (1994) list a number of cosmetics and toiletry additives simply as "extracts" without further detail of their chemical composition. Extracts of corn germ, barley, wheat, wheat germ, oats and oat bran are produced by a number of companies and used in a range of products.

2.2.11 Ethanol

Ethanol, also called ethyl alcohol, is used in the manufacture of essential oils, flavourings, perfumes, hard and soft beverages, pizza crusts, printing inks, soaps, vinegar and pharmaceuticals (Secondini, 1990; Lewis, 1989). It is also utilized as an antimicrobial agent and solvent.

2.2.12 Polymers

A number of derivatives from cereal plants have application in biodegradable polymers (Forward, 1994). An example is pullulan which may be derived from corn products using the fungus *Aureobasidium* (Leathers and Gupta, 1994). Pullulan can be made into films, nylon-like fibres and polystyrene-like compression mouldings that have application in food, pharmaceuticals, cosmetics and other industries. The world market for pullulan is approximately $12 million U.S. and at present this is produced by starch fermentation (Dr. T. Leathers, personal communication).

2.2.13 Calcium Magnesium Acetate

Calcium magnesium acetate, derived from corn, is used for de-icing on bridges (Forward, 1994).

2.2.14 Xylitol

Xylitol is a sweetener that is primarily produced in Finland using acid-treated fibres of birch wood (Chemical Marketing Reporter, 1993). It does not cause dental caries and is used in chewing gums. According to Dr. Timothy Leathers at USDA, there may be potential for expanding the current world market of $28 million U.S. to include use in foodstuffs for diabetics. Xylitol can be made from chemical conversion of the xylan found in corn fibre. A number of major companies in the U.S. have looked at the commercialization of this technology.

2.2.15 Aquaculture Feed Products

Dr. Victor Wu and his associates at the Biopolymer Research Unit, National Center for Agricultural Utilization Research (USDA) have evaluated the use of corn gluten feed in Nile tilapia diets as a low cost replacement for conventional catfish feed. As a result, corn gluten meal is now being used on a commercial scale in the industry in conjunction with Arthur Daniels Midland (Dr. T. Leathers, personal communication).

2.2.16 Cinnamic Acid

Different forms of cinnamates have use in sunscreens and in the manufacture of esters for perfumes, medicines and glass prisms and lenses (TWG Consulting Inc., 1995; Merck Index, 1976).

Cinnamates are found in wheat bran.

2.2.17 Alternan

USDA scientists have shown that corn condensed distillers' solubles can be utilized as a nutrient source for the production of alternan, a new bacterial gum that can be used in place of gum arabic in food products (Dr. T. Leathers, personal communication).

2.2.18 Diethylamine

Found in barley kernels, diethylamine has use in the rubber and petroleum industry, and in flotation agents, resins, dyes and pharmaceuticals (Merck Index, 1976). It is generally manufactured from ethanol and ammonia.

2.2.19 Piperidine

Piperidine has been isolated from barley (Le Jardin de l'Aigle Reg., 1994) and is used in the pharmaceutical industry as an antihistamine (drug facts and comparisons, 1994).

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Return to Coproducts and Near Coproducts of Fuel Ethanol from Grain, Table of Contents

Chapter 3. Current Research Efforts into Coproducts and Near Coproducts formed when Grain is Fermented to produce Ethanol

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1. RESEARCH

1.1 Introduction

A survey of the recent scientific literature as well as discussions with a number of people involved in the fuel ethanol industry has indicated that there is a significant amount of research being conducted on coproducts and near coproducts of fuel ethanol fermentation. This represents a major change from the situation reported by Monenco AGRA (1993) when coproduct research was felt to have been relatively meagre. While some of the coproduct research is centred at university and government laboratories, a great deal is being done collaboratively with industry. For this reason, one inevitably runs into the question of confidentiality, which is naturally important to any commercial venture. In some cases, companies would merely acknowledge that they are doing research while in others a brief outline of their project(s) were available.

Information on current research areas can be found from a variety of sources. Conference proceedings such as those from the Biomass Conferences of the Americas, Corn Utilization and Wheat Utilization Conferences contain abstracts and summaries of presentations. Research institutions such as the Research Branch of Agriculture and Agri-Food Canada or the United States Department of Agriculture, Agricultural Research Service often publish summaries of their research activities. Another important source of information is that of on-line databases which list research projects. Examples include the Inventory of Canadian Agri-Food Research (ICAR), Current Research Information System (CRIS, U.S.), Australian Rural Research in Progress (ARRIP), Agricultural Research Projects (AGREP, European Union) and Crop Association Sponsored Research Archive (CASRA, U.S.). Patent information may also prove useful.

In addition to research being done specifically on grain-to-ethanol process byproducts, there is the whole area of value-added cereal components or near coproducts that is not necessarily connected to the ethanol industry but is still of significance as the industry may provide a low-cost source of raw material for extraction. This leads one to the concept of a biorefinery which incorporates ethanol production with grain preprocessing technology, chemical extraction using ethanol and/or carbon dioxide produced in plant, and microbial conversions to produce a wide range of products for food and non-food industries.

Discussions with a number of people in the ethanol industry in North America indicated that grain biorefineries appear to be a way of the future. Similarly, in Europe, a project entitled "The Whole Crop Biorefinery Project" is being run under the auspices of the European Collaborative Linkage of Agriculture and Industry through Research (Christensen, 1994). This project involved ten research institutions and industrial partners from five European nations and covered six topic areas, five of which involved wheat straw and kernels.

A large new area of research is that of functional foods (also called nutraceuticals, designer foods, pharmafoods, phytochemicals or medical/medicinal foods) (Food Focus, 1995)). The functional food concept began in Japan and is well established there. Currently eleven categories of functional foods are recognized in Japan for specific health use. The largest areas are dietary fibre and oligosaccharides. Interest is growing in Europe and North America and there have been numerous conferences, symposia and workshops on this topic in recent years.

The cereal grains have been investigated to some extent to elucidate their nutraceutical properties. A recent study by Food Focus (1995) for Agriculture and Agri-Food Canada examined the status of the functional food industry in Canada. Of 35 companies surveyed, a number were producing plant products such as fibre, that they believed to have nutraceutical qualities. Because coproducts of fuel ethanol fermentation may provide a source of material for further development into functional foods, a number of research projects and recent discoveries pertaining to this field have been identified in this report.

The development of the functional food or nutraceutical industry in Canada is limited at present by a number of factors including the Canadian government's *Food and Drugs Act and Regulations* (Food Focus, 1995). If a health claim is made for a product, it is then classified as a drug by Health Canada and requires rigorous clinical testing for safety, efficacy and usage (Micheline Ho, personal communication). Health Canada has received requests for information in regards to classification of different cereal derivatives as functional foods. Details of actual submissions are treated as confidential. Health Canada is presently considering the need for a different means of classification for foods which have proven health benefits.

1.2 Research in Canada

1.2.1 Industry

Canamino Inc. produces value-added oat derivatives for use in cosmetics and over-the-counter pharmaceuticals. They use a patented process developed by Dr. Dave Paton and Dr. Bill Collins of Agriculture and Agri-Food Canada to separate the bran and flour portions of the oat groat. At present, Canamino products are being used around the world by the major household cosmetic and toiletry firms including Avon, Jergens, Estée Lauder and Cheeseborough Ponds. They are also having discussions with a number of the so-called "green" cosmetics firms. Research by Canamino is ongoing at their Nepean, Ontario facility.

Ceapro Developments Inc. of Edmonton, Alberta is active in the functional food area (Pilip, 1995). Working with government research and development facilities, they have developed a number of products containing dietary oat fibre including a Japanese noodle, a type of yoghurt based on fermented oat porridge and a health drink.

Kilborn Inc. was involved in the development of the Multiple Oxygenate Products (MOP) process for the production of ethanol from corn, other grains or other starchy or cellulosic compounds. This process generates an number of coproducts including distillers' dried grains with solubles (DDGS), methanol, ethyl t-butyl ethers (ETEB) and methyl t-butyl ether (MTBE). More information about the MOP process can be found by reaching Gerry Hamaliuk at Kilborn Inc. in Toronto.

Mohawk Oil Company Ltd. recently announced a $1.5 million upgrade of their plant in Manitoba

to produce a fibre and protein coproduct called Fibrotein, for use as a food additive for the Canadian market (Rampton, 1995). Mohawk is using technology owned in the USA by Cereal Ingredients Inc. (Dr. E. St. Denis, personal communication). It has taken Mohawk Oil approximately three years to acquire the Canadian rights, undertake research and feasibility studies and get Health Canada approval. Initially 1.5 million kg/annum of the product will be produced with potential expansion to more than three times that amount. Higher quality wheat will be required as a feedstock. According to Mohawk Oil, production of Fibrotein will increase the economic feasibility of fuel ethanol production.

POS Pilot Plant Corporation has been involved in research and development activity for the past 20 years (Dr. Paul Fedec, personal communication). POS is a private, non-profit company serving the agri-food industry and has 48 industrial, associate and government members. Ninety percent of POS (Protein Oil Starch) activity is done on a fee for service basis for industry, both in Canada and the United States. POS has the capacity to wet process cereals including wheat, corn, barley and oats, for fractionation, separation and isolation of protein, starch and minor components. The Corporation also has equipment to modify extracts by extrusion, drying, etc. POS Pilot Plant Corporation has recently added a wholly-owned commercialization arm to carry basic research through to full scale production. In the past, POS worked with Canamino to develop commercial oat-based products based on research conducted at Agriculture and Agri-Food Canada.

TDI Projects Inc. of Edmonton, Alberta is active in the area of value-added wheat processing. They are involved in the technical aspects of a plant to be constructed at Red Deer, Alberta as well as in the construction of a small plant in Washington State for product development. This latter plant will refine process technology and produce food fibre products for testing and market trials.

Tkac and Timm Enterprises Ltd. has developed a value-added wheat fibre product for the human food market called PRIMAFIBRE (Tkac and Timm, 1995). Their patented technology involves the sequential removal of the bran layers from the wheat kernel leaving a starch enriched grain that could then be fermented to form fuel ethanol. PRIMAFIBRE is available in a range of particle sizes and can be used in a variety of food products including breakfast cereals, health foods, bakery products, snack foods and cookies. The Tkac and Timm process lends itself to use in a biorefinery with a number of product streams including ethanol, wheat germ, Vitamin E (extracted from wheat germ), various bran products and other chemicals derived from the bran fractions by further refining (TWG Consulting Inc., 1995).

1.2.2 University

Dr. Mike Ingledew and his group at the Applied Microbiology and Food Science Department, **University of Saskatchewan** are researching the use of very high gravity fermentation to produce ethanol. In this process, now at the pilot scale testing stage, the grain can be dry milled before cooking and fermentation to yield a bran fraction which has applications in the food industry (Bioenergy West, 1994). Grain solids (mostly bran) removal after solubilization, of starch but before fermentation, creates an opportunity for production of value-added products.

Dr. R.S. Bhatty at the **University of Saskatchewan** is involved in a collaborative project entitled "Promotion of hulless barley in food and industry". He and his coworkers are looking at value-added products including ß-glucan and bran.

Dr. John Postlethwaite and Dr. S. Rohani at the **University of Saskatchewan** were involved with an

innovative design project done by a group of four undergraduate students in 1995 (Eggum et al., 1995). This project looked at the application of extraction technology in an integrated ethanol plant and feed-lot operation. Using technology developed at the University of Saskatchewan, it was possible to produce a wheat concentrate for human food use instead of the traditionally produced and lower value wet distillers' grains for livestock consumption. The wheat concentrate was of high protein and fibre content, had low energy and an acceptable flavour.

Extractive fermentation, being developed at **Queens University** by Dr. Andrew Dauglis and his associates, yields a feed byproduct that includes dried yeast from the fermentation and dehydrated spent fermentation medium, as well as distillers' dried grains. It is at the pilot scale testing stage.

The Alberta Barley Commission is sponsoring a project looking at the functional properties of barley ß-glucan with a long term view to developing a barley biorefinery which would produce a number of value-added products. Dr. F. Temelli at the **University of Alberta**, Faculty of Agriculture, Forestry and Home Economics is in charge of this study. Her group has isolated and analyzed an enriched fraction (up to 78%) of ß-glucan from barley. The next step will be evaluation and development of the product for potential food usage. Dr. Sam Jadhav of the Alberta Department of Agriculture Food and Rural Development in Leduc, Alberta, is the project leader in a co-study evaluating techniques to separate the starch, beta-glucan and protein fractions of the barley grain and to assess their functional properties.

1.2.3 Government

The Centre for Food and Animal Research, Agriculture and Agri-Food Canada in Ottawa is looking at potential non-food uses of cereal crops as well as extraction of minor components for nutraceutical or functional food usage. There are currently opportunities for collaborative projects to develop niche markets for specific commodities. Some of the projects being conducted with industrial partners are listed below:

Dr. John Mullin is working with an industrial partner in order to characterize the bran fractions that they are able to remove from wheat kernels using patented technology. They are interested in determining contents of soluble and insoluble fibre, phytate, starch, oligosaccharides, and solvent extractable phenolics of the different bran layers.

Dr. Shea Miller is working with an industrial partner to investigate the distribution of ß-glucan in the grains of a number of different oat cultivars with a view to utilizing them for different processing functions.

Dr. Peter Wood is also studying ß-glucan in oats. In conjunction with industry and clinicians he is looking at improving the potential for producing high beta-glucan products for commercial use.

Dr. Bill Collins is involved in the evaluation of phenolic avenanthramides in oats and wheat. Particular emphasis has been on the bound forms of the hydroxycinnamic acids found in oat hulls and groats. He is also investigating the presence of flavonoids in wheat germ. Flavonoids are known to have antioxidant and antitumor activity. In another study, Dr. Collins and his coworkers are looking at the sterol esters found in wheat germ using unique extraction methods.

Dr. Dave Paton at Research Branch, Agriculture and Agri-Food Canada in Saskatoon is working on oats and oat components, identifying and evaluating chemical and functional properties and

developing appropriate technology to produce value-added products from oats.

1.3 Research in the United States

1.3.1 Ethanol Coproduct Research

Both the Agricultural Research Service of the United States Department of Agriculture (USDA-ARS) and the U.S. Department of Energy are involved in fuel ethanol research in the United States. The Department of Energy runs its program through the Office of Energy Efficiency and Biofuels Information Network (BIN).

USDA-ARS has a biofuels research program looking at the use of renewable agricultural feedstocks for the production of fuels and value-added coproducts. They are trying to find solutions to the lack of value-added coproducts currently in the marketplace, and the associated high cost of recovery and separation of potential coproducts that have been identified.

USDA-ARS separates coproducts into three categories - high, substantial and moderate value products. Examples of high value products include medical and veterinary pharmaceuticals; substantial value products include environmentally friendly pesticides, biodegradable plastics, edible films, industrial enzymes and food additives; moderate value products include bulk chemicals and intermediates such as acetate, glycerol, lactate and polyalcohols. Research priorities at USDA-ARS include development of composite materials for non-food uses, non-food uses of protein coproducts, molecular modelling of polymers produced from coproducts and bioconversion of residual carbohydrates (Leathers et al., 1992). Programs and research team leaders located at three USDA-ARS Centres are listed below.

1. New Process Operations and Systems for Refining and Converting Grains to Value Added Products (Wheat)
George Robertson, Western Regional Research Center, Albany, California
(501) 559-5866

2. New Processes for Generating Valuable Coproducts from Corn Fibre
Kevin Hicks, Eastern Regional Research Centre, Wyndmoor, Pennsylvania
(215) 233-6579

3. Value Added Coproducts from Biofuel Conversion
Richard Greene, National Centre for Agricultural Utilization Research, Peoria, Illinois
(309) 685-4011

A major study of recently completed and ongoing research projects on corn-to-ethanol production and coproducts has recently been completed by Dr. Nicholas Powers (1995) of Powers Agribusiness Research, Shaker Heights, Ohio. This study was funded by USDA-ARS and the Illinois Department of Energy and Natural Resources and will be available from the principal investigator, Brian Donnelly, at University Park, Southern Illinois University at Edwardsville. It is part of a larger study to consider the feasibility of establishing a pilot plant for corn-to-ethanol production in order to determine the commercial potential of processes that have been found successful in the laboratories of government, universities and industry.

In his report, Powers presents summaries of 103 projects related to corn-to-ethanol production

including, in more detail, those located at the USDA-ARS Regional Research Centres already mentioned. Research projects located in the U.S. concerned with coproduct production are listed below giving title, principal investigator, location, telephone number and a brief statement of research as it concerns coproducts. A great deal of interest is apparent in the development of techniques to ferment corn fibre and derive both greater ethanol yields and higher value coproducts. These projects have been listed separately under the heading, corn fibre. For more information about the different research projects, interested parties should refer to the report itself or contact Brian Donnelly.

WHEAT

1. Refining of Wheat to Value-Added, Purified Components and Ethanol
Dr. George Robertson1 and Ralph Kurtzman, 1Western Regional Research Center, Albany, California
Tel: (510) 559-5866
(Preprocessing of wheat to produce high quality protein coproducts)

CORN

1. Genetic Engineering of Specialized Corn Hybrids with Value-Added Grain Characteristics
Dr. Torbert Rocheford, University of Illinois at Urbana-Champaign
Tel: (217) 333-3420
(Development of corn hybrids with high-oil content, altered fatty acid composition and/or high-starch content for increased or novel coproduct production)

2. Molecular Genetic Modification of Lysine Synthesis in Corn
Dr. Burle Gengenbach, University of Minnesota
Tel: (612) 625-6282
(Increased quality and quantity of amino acids in coproducts)

3. Value-Added Coproducts in Ethanol Production by the Sequential Extraction Process
Dr. Lawrence Johnson1, Dr. Mila Hojilla-Evangelista, Dr. Deland Myers and Dr. Anthony Pometto III, 1Iowa State University
Tel: (515) 294-4365
(Improving the yield and quality of coproducts, including protein, fibre and oil, produced using the Sequential Extraction Process)

4. Simultaneous Corn Oil Extraction and Alcohol Dehydration, Protein Extraction, and Enzymatic Hydrolysis of Corn Starch after Extractions
Dr. Li-Fu Chen, Purdue University
Tel: (317) 494-8263
(Increased cost-efficiency via corn oil and edible protein extraction)

5. Germ Recovery Process for Dry Grind Corn
Dr. Steve Eckhoff, University of Illinois at Urbana-Champaign
Tel: (217) 244-4022
(Development of technology to extract germ from corn kernels)

6. Pervaporation of Acetone, Butanol, Ethanol

Dr. Michael Meagher, University of Nebraska-Lincoln
Tel: (402) 472-2342
(Development of process to extract acetone, butanol or ethanol from dilute fermentation broth using membrane technology)

7. Flavorzyme
Mr. Neal Briggi, Novo Nordisk Entotech, Inc.
Tel: (203) 790-2600
(Use of a new protease enzyme to effect hydrolysis of corn proteins into amino acids that can then be used in the manufacture of meat flavours)

8. Lypase
Mr. Neal Briggi, Novo Nordisk Entotech, Inc.
Tel: (203) 790-2600
(Use of a new enzyme to break down fat or oil molecules into new fats or oils with desired characteristics)

9. New Protein Coproducts from Corn Milling
Dr. Leland Dickey, Eastern Regional Research Center, Philadelphia, Pennsylvania
Tel: (215) 233-6640
(Economically feasible technology for deriving a zein-enriched protein product)

10. Protein-Based Coproducts
Dr. Victor Wu, The National Center for Agricultural Utilization Research, Peoria, Illinois
Tel: (309) 681-6377
(Development of uses for corn protein products beyond the feed industry, e.g. zein)

11. Adding Value to Corn Proteins
Dr. Munir Cheryan, University of Illinois at Urbana-Champaign
Tel: (217) 333-9332
(Use of enzymes and membranes to develop corn protein products for human foods)

12. Higher-Value Corn Proteins
Mr. Sammy Pierce, EnerGenetics, Keokuk, Iowa
Tel: (217) 453-2340
(Production of undenatured corn protein for use in the food industry, using grinding and membrane techniques)

13. Incorporating Protein and Carbohydrate Residues into Composite Materials
Dr. Richard Greene1, Dr. S. Imam, Dr. Victor Wu, 1The National Center for Agricultural Utilization Research, Peoria, Illinois
Tel: (309) 681-6377
(Use of corn proteins and carbohydrates in the production of industrial biopolymers and composite materials)

14. Converting Residual Carbohydrates to Value-Added Coproducts
Dr. Timothy Leathers, The National Center for Agricultural Utilization Research, Peoria, Illinois
Tel: (309) 681-6377
(Production of pullulan, astaxanthin and xylitol from corn fermentation byproducts using fungi, yeast

and yeast-like fungi)

15. Value-Added Products from Steep Water and Residual Fiber of Corn Wet Milling Processes
Dr. George Tsao, Purdue University
Tel: (317) 494-4068 or 494-7022
(Evaluation of adsorption to remove lactic acid, phytic acid, amino acids and other value-added coproducts from steep water)

16. Separation of Glycerol and Organic Acids in Model Ethanol Stillage by Electrodialysis and Precipitation
Dr. Munir Cheryan1 and Dr. Sarad Parekh, 1University of Illinois at Urbana-Champaign
Tel: (217) 333-9332
(Evaluation of the potential for separating and isolating glycerol and organic acids (eg. succinic and lactic acids) from ethanol stillage using electrodialysis and selective crystallization)

17. A Pilot Scale Conversion of Lactic Acid, Glycerol, and Residual Sugars and Proteins from the Thin Stillage of Starch Fermentations to Yeast Single Cell Protein
Mr. Bob Lehman and Dr. Clark Dale1, 1Bio-Process Innovation, Inc., West Lafayette, Indiana
Tel: (317) 494-1195
(Production of yeast single cell protein from components of corn thin stillage)

18. Production of Propionic and Acetic Acids by Extractive Fermentation
Dr. Bonita Glatz1 and Dr. Charles Glatz, 1Iowa State University
Tel: (515) 294-3970
(Development of extractive fed-batch fermentation with immobilized cells which allows for cost-effective recovery of propionic and acetic acids)

19. Chemicals from Starch and/or Byproducts of Corn-to-Ethanol Production
Dr. Dick Antrim, Genencor International, Inc., Cedar Rapids, Iowa
Tel: (319) 368-7602
(Use of genetic engineering techniques to create new enzymes for conversion of starch and byproducts into chemicals for both food and non-food industries)

20. Recovering Glycerol
Mr. Gary Welch, Pekin Energy Company, Pekin, Illinois
Tel: (309) 347-9271
(Development of effective technology for the extraction of glycerol from fermentation byproducts)

21. Carbon Dioxide and Yeast Cell Utilization
Dr. Li-Fu Chen, Purdue University
Tel: (317) 494-8263
(Use of super critical CO_2 extraction for recovery of food-grade protein from yeast/Use of CO_2 as a sterilant for food and pharmaceutical products)

22. Yeast Invertase as a Coproduct of Continuous Ethanol Fermentation
Dr. Li-Fu Chen, Purdue University
Tel: (317) 494-8263
(Production of extracellular invertase by *Saccharomyces uvarum* using media containing corn steep

liquor)

23. Electrochemical Reduction of Carbon Dioxide to Fuels
Dr. Dan DuBois, National Renewable Energy Laboratory, Golden, Colorado
Tel: (303) 384-6171
(Conversion of CO2 to methanol using specially developed catalysts)

24. Bioconversion of Carbon Dioxide, the Major Byproduct of Fermentation, to Ethanol
Dr. F. Tabita1 and Dr. T. Conway, 1The Ohio State University
Tel: (614) 292-4297
(Conversion of CO2 to ethanol using genetically engineered CO2-fixing bacteria with cloned ethanol production genes)

25. Corn-Based Fish Feeds
Dr. Victor Wu, Dr. R. Rosati, Dr. David Sessa1, Dr. P. Brown, 1National Centre for Agricultural Research, Peoria, Illinois
Tel: (309) 681-6351
(Development of low cost talapia feeds containing corn-to-ethanol fermentation byproducts including gluten meal, distillers' grains with solubles and gluten feed)

26. Producing Feeds and Developing New Products from the Coproducts of Wet Corn Milling
Dr. Robert Friedman, American Maize Products Company, Hammond, Indiana
Tel: (219) 659-2000
(Use of corn fibre, germ, oil and protein for the production of value-added materials for food and non-food use)

27. Feeds and New Products from the Coproducts of Wet Corn Milling
Mr. Dick Roberts, CPC International, Summit-Argo, Illinois
Tel: (708)563-6706
(Development of new feed, food and other industrial products from corn fibre and protein, including zein)

28. Stillage Clarification with Membranes
Mr. Gary Welch1 and Dr. Munir Cheryan, 1Pekin Energy Company, Pekin, Illinois
Tel: (309) 347-9271
(Development of membrane technology to separate water from insolubles in stillage)

29. Membranes
Dr. John Long, Archer Daniels Midland Company, Decatur, Illinois
Tel: (217) 424-5399
(Development of membrane technology for production efficiency and recovery of coproducts)

CORN FIBRE

1. Alkali Wet Milling of Corn
Dr. Steve Eckhoff, University of Illinois at Urbana-Champaign
Tel: (217) 244-4022
(Development of a purer and therefore higher-value corn fibre coproduct)

2. Producing Organic Acids from Corn Gluten Feed and Other Plant Biomass
Dr. Shang-Tian Yang, The Ohio State University
Tel: (614) 292-6611
(Conversion of cellulose and hemicellulose fractions of corn gluten meal to organic acids including acetic, propionic, butyric and lactic acid)

3. Value-Added Products from Steep Water and Residual Fiber of Corn Wet Milling Processes
Dr. George Tsao, Purdue University
Tel: (317) 494-4068 or 494-7022
(Fungal fermentation of corn fibre to produce lactic acid)

4. Converting Corn Fiber to Lactic Acid
Dr. George Tsao, Purdue University
Tel: (317) 494-4068 or 494-7022
(Fermentation of corn fibre to value-added coproducts, including lactic acid, using fungi)

5. New Processes for Generating Valuable Coproducts from Corn Fiber
Dr. Robert Moreau, Eastern Regional Research Center, Philadelphia, Pennsylvania
Tel: (215) 233-6428
(Development of technology for extraction of lipids and polysaccharides from corn fibre which have potential use in food, pharmaceutical and other industries)

6. Improved Feedstocks for Biofuels
Dr. Badal Saha1 and Dr. Rodney Bothast, 1National Centre for Agricultural Utilization Research, Peoria, Illinois
Tel: (309) 685-6276
(Pretreatment of corn fibre and evaluation of a number of yeast strains to broaden the feedstock base that can be converted to ethanol and other potential products)

7. Novel Ethanol Conversion Technologies for Lower Biofuel Cost
Dr. Robert Hespell and Dr. Rodney Bothast1, 1The National Center for Agricultural Utilization Research, Peoria, Illinois
Tel: (309) 681-6566
(Development of new microbial strains that can convert corn fibre to ethanol with a potential coproduct stream including biodegradable polymers, deicers from acetic acid and food additives)

8. Novel Systems for High-Level Expressions of Fungal Products
Dr. Shelby Freer and Dr. Rodney Bothast1, 1The National Center for Agricultural Utilization Research, Peoria, Illinois
Tel: (309) 681-6566
(Conversion of corn fibre to ethanol and a number of value-added coproducts including acetic acid and CO2)

9. Arabinose-Fermenting Yeasts
Dr. Thomas Jeffries, USDA, Madison, Wisconsin
Tel: (608) 231-9456
(Development of arabinose-fermenting yeasts which convert part of the corn fibre to ethanol thus

affecting the quantity and quality of coproducts produced)

10. Xylose-Fermenting Yeasts
Dr. Thomas Jeffries, USDA, Madison, Wisconsin
Tel: (608) 231-9456
(Development of xylose fermenting yeasts which convert part of the corn fibre to ethanol thus affecting the quantity and quality of coproducts produced)

11. Genetic Engineering of Bacteria for Fuel Ethanol Production from Biomass
Dr. Lonnie Ingram, University of Florida
Tel: (904) 392-5924
(Project includes development of bacteria that may have the ability to ferment corn hulls and fibres and thereby affect the quantity and quality of byproducts)

12. Pretreatment, Hydrolysis, and Fermentation of Corn Fiber
Dr. Bruce Dale1 and Dr. Rodney Bothast, 1Texas A&M University
Tel: (409) 845-3413
(Fermentation of hexoses and pentoses in corn fibre leaving a higher quality coproduct for feed purposes)

13. Corn Fiber Coproducts Derived from Production of Biofuels
Dr. Michael Ladisch, Purdue University
Tel: (317) 494-7022
(Increased content of protein in DDGS and gluten feed by removal of cellulosic fraction of corn fibre)

14. Ethanol Production from Corn Fiber, Paper, and Other Biomass Materials
Dr. Jonathan Mielenz, National Renewable Energy Laboratory, Golden, Colorado
Tel: (303) 275-4489
(Use of corn fibre to produce ethanol and other coproducts including protein)

15. Converting Corn Fiber to Ethanol
Dr. Ting Carlson, Cargill Inc., Minneapolis, Minnesota
Tel: (612) 742-6508
(Production of ethanol and other products from corn fibre using acids, enzymes, yeasts, fungi and bacteria)

16. The Breeding of Pentose Fermenting Yeast Strains for Bioenergy Production
Dr. Roy Thornton, Indiana University
Tel: (317) 455-9290
(Development of a strain of the yeast *Pachysolen tannophilus* that can convert pentose (hemicellulose) and hexose (glucose) sugars to ethanol, yielding feed products with a higher protein content)

1.3.2 Food Use Research

1. Improving the Nutritional and Health Promoting Properties of Cereal Foods
Dr. W.H. Yokoyama, Dr. T.S. Kahlon and Dr. B.E. Knuckles, Western Regional Research Center, Albany, California 94710

(Development of value-added cereal grains such as wheat, oats and barley, that contain compounds, such as beta-glucan, that has been linked to lower risk of heart disease and other chronic diseases)

2. Processing and Alternate Uses of Hard Red and Hard White Winter Wheats
Dr. C.F. Klopfenstein and Dr. C.E. Walker, Kansas State University, Manhattan, Kansas 66506
(Investigation of recovery and utilization of wheat fibre for use in human food products)

3. A Novel Continuous Production of Value-Added Food Additive, Xanthan Gum, from Corn Products
Dr. D.B. Min and Dr. S.T. Yang, Dept. of Food Science & Technology, Ohio State University, Columbus, OH. 43210
(Cost-effective production of xanthan gum from corn steep liquor and corn using a novel continuous fermenter)

4. Value-Added Wheat Products
Dr. R.R. Hahn and Dr. G. Brester, Grain Science and Industry, Kansas State University, Manhattan, Kansas 66506
(Identification, evaluation, development, technological transfer and market assessment of value-added opportunities for wheat products)

5. Development of New Oils, Starches and Antioxidants from Soybean, Corn and Oat
Dr. P.J. White, Family & Consumer Science, Iowa State University, Ames, Iowa 50011
(Study includes development of new oils with unique fatty acid compositions and investigation of the antioxidant potential of naturally occurring compounds from oat)

6. Biomass Refining of Wheat to Value-Added Food Products, Non-Food Products, Chemicals and Ethanol
Dr. G.H. Robertson, Western Regional Research Center, Albany, California 94710
(Evaluate of the utility of integrating the process of post-fermentation ethanol dewatering [by feedstock-grain-based adsorption] with the process of pre-fermentation component separation [by extraction of non-starch components of wheat using ethanol])

7. New Process Operations and Systems for Refining and Converting Grains to Value- Added Products
Dr. G.H. Robertson and Dr. R.H. Kurtzman, Jr., Western Regional Research Center, Albany, California 94710
(Identification and evaluation of potential systems to fractionate grain into component fractions for value-added usage)

8. Enzymatic Modification of Soybean and Wheat Proteins for Food and Non-Food Products
Dr. F.F. Shih and Dr. P.J. Wan, Agricultural Research Service, Southern Regional Research Center, New Orleans, Louisiana 70179
(Investigation of enzymatic methods to modify soybean and wheat proteins for the development of functional properties desirable in new and improved food and non-food products)

1.3.3 Industrial Use Research

1. Nonedible Wheat Gluten Films for Use as Mulch and Bags
Dr. V.M. Ghorpade; Dr. C.L. Weller, Biological Systems Engineering, University of Nebraska,

Lincoln, Nebraska 68583
(Design of high-strength and low-solubility biopolymer films from wheat gluten, characterization of molecular interaction between polymers during film forming processes and study of compostability of cast films)

2. Production of a Corn-Based, Commercially-Emerging Gum Using Cell Immobilization
Dr. T.P. West, Biochemistry, South Dakota State University, Brookings, South Dakota 57007
(Production of pullulan by mutant cells of the fungus *Aureobasidium pullulans* using immobilized cells on corn syrup)

3. Bioconversion of Ethanol Production Byproducts into Acetate (CMA)
Dr. W.R. Gibbons, Biology & Microbiology, South Dakota State University, Brookings, South Dakota 57007
(Use of low-value ethanol production byproducts, such as thin stillage, CO2 and corn steep liquor, for fermentative production of acetate using the thermophilic anaerobe *Clostridium thermoaceticum)*

4. CMA from Corn: Scale-Up of the Fermentation and Recovery Processes
Dr. M. Cheryan and Dr. S. Parekh, Food Science, University of Illinois, Urbana, Illinois 61801
(Scale-up and optimization of calcium-magnesium acetate (CMA) production in batch, fed-batch and/or continuous membrane bioreactors using selected membrane technologies to de-water, recover and concentrate CMA/acetate from the fermentation broth)

5. Modifications of Cereal Starches, Dextrins, and Cycloamyloses and Their Derivatives for New Uses
Dr. J.A. Rendleman, Dr. J.M. Gould and Dr. H.L. Griffin, Northern Regional Research Center (USDA), Peoria, Illinois 61604
(Graft/crosslink alpha-glucans with beta-glucans or related biopolymers and evaluate commercial potential of product for industrial use)

2. LIST OF REFERENCES

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Appendix 1. List of Contacts

The following people or organizations were contacted by telephone, facsimile or letter during the course of this project:

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Coproducts of Fuel Ethanol Production

Les coproduits de la production d'éthanol

Reference Database

As part of the Canadian Green Plan Ethanol Program, Starchy Waste Streams Evaluation Project, administered by Agriculture and Agri-Food Canada, a reference database dealing with the coproducts of ethanol fermentation from grain, has been developed. The purpose of this database is to aid researchers in becoming more familiar with the current state of coproduct research when corn, wheat, barley or oats are used as fermentation feedstocks for the production of fuel ethanol.

The data-base is organized into five sections including general papers, and those pertaining more specifically to barley, corn, oats or wheat. For more information, please contact Chris Tibelius at (613) 224-9988.

Base de données de référence

Dans le cadre du Projet d'évaluation des fractions résiduaires féculentes, une base de données sur les coproduits de la dégradation des céréales en éthanol a été élaborée. Ce projet est l'un des volets du Programme Éthanol (Plan vert du Canada) régi par Agriculture et Agroalimentaire Canada. L'objectif est de familiariser les scientifiques avec les derniers développements de la recherche sur les coproduits obtenus lorsque l'on fermente certaines céréales - blé, avoine, maïs et orge - pour en tirer un biocarburant, l'éthanol.

La base de données se divise en cinq sections. La première contient des articles d'intérêt général et les quatre autres, plus spécialisées, portent sur l'avoine, l'orge, le blé, et le maïs respectivement. Pour de plus amples renseignements, communiquez avec Chris Tibelius au (613) 224-9988.

General Papers | Barley | Corn | Oats | Wheat
Articles d'intérêt Général | Orge | Maïs | Avoine | Blé

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M. Jomphe, Web Design

Appendix 3. Additional References

The production of a reference data-base is a continuous process as newly discovered citations lead to other papers and new scientific findings are reported in the literature. A number of references came to light after the preparation of the summary of the literature and the reference data-base for the Agriculture and Agri-Food Canada Electronic Information System (ACEIS). These papers are recorded in this appendix and will be added to ACEIS when and if an opportunity for an update occurs.

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General Papers | Barley | Corn | Oats | Wheat
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Barley / Orge

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Corn / Maïs

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Oats / Avoine

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